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The Journal *of the* Society of Dyers and Colourists

Volume 75 Number 11

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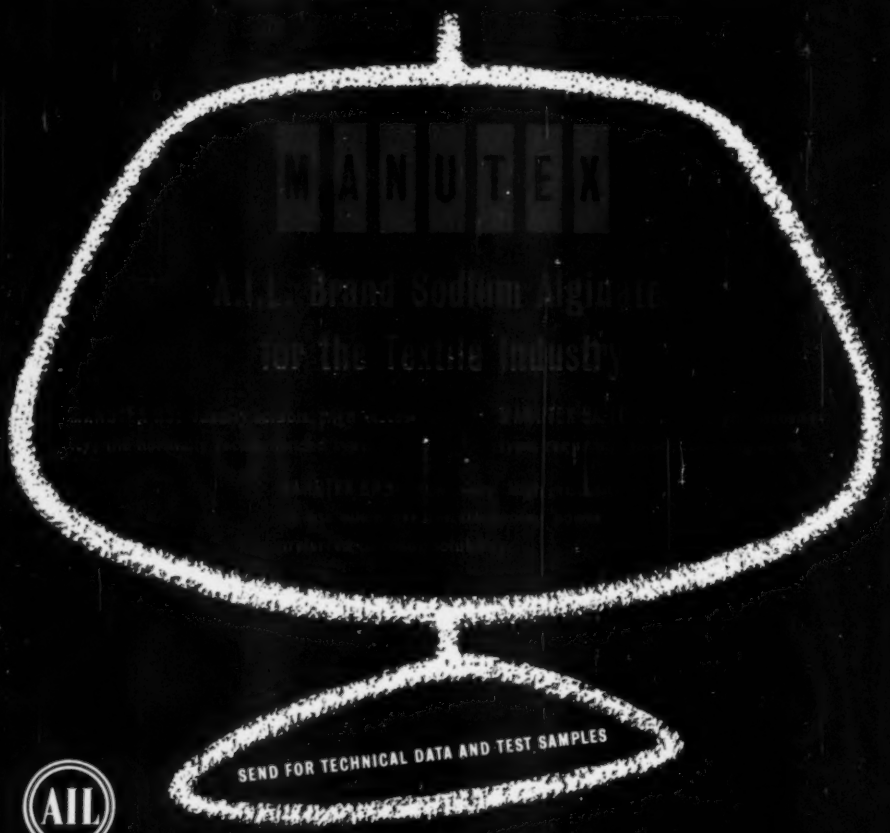

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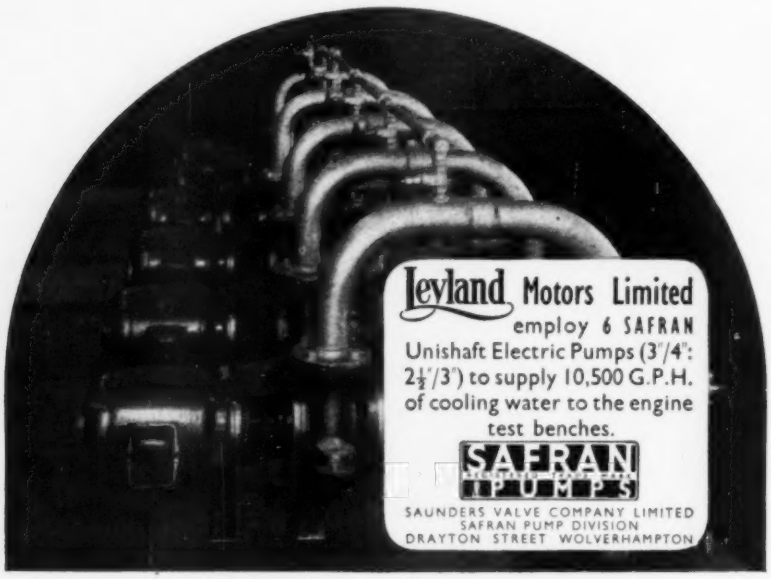

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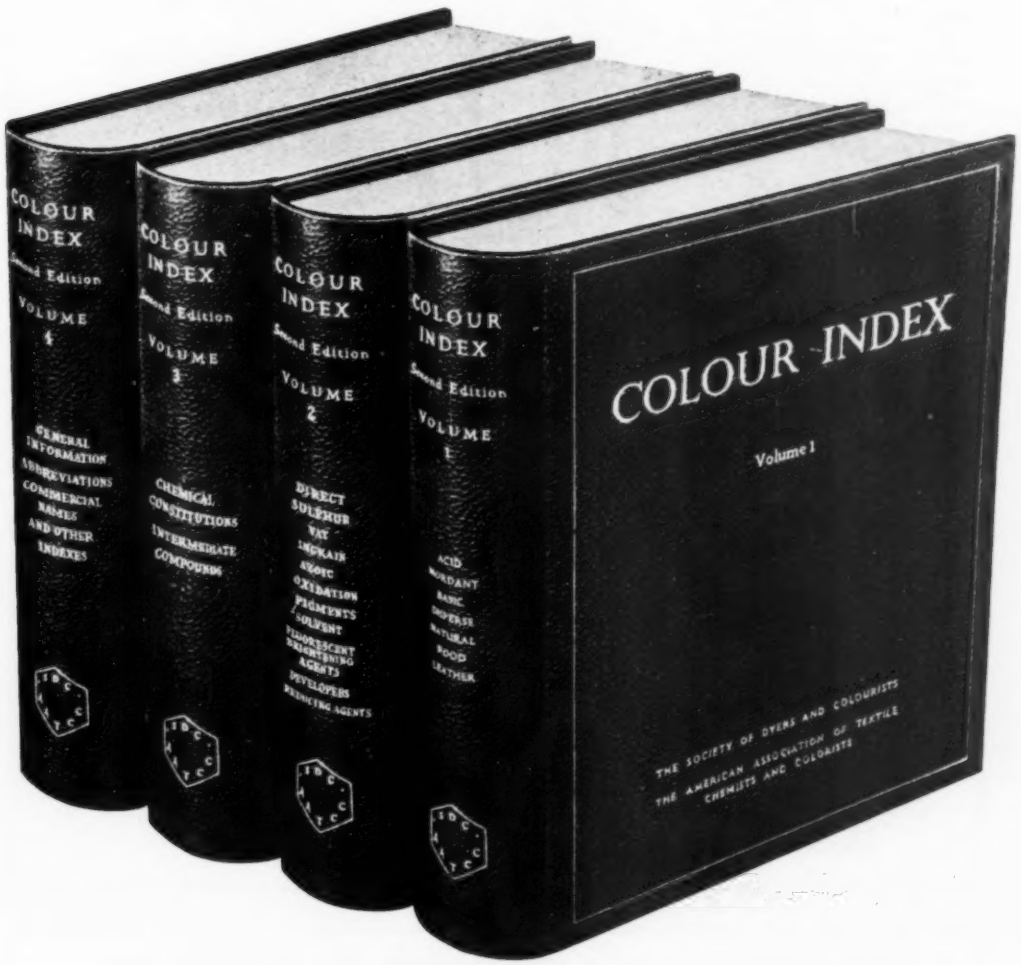

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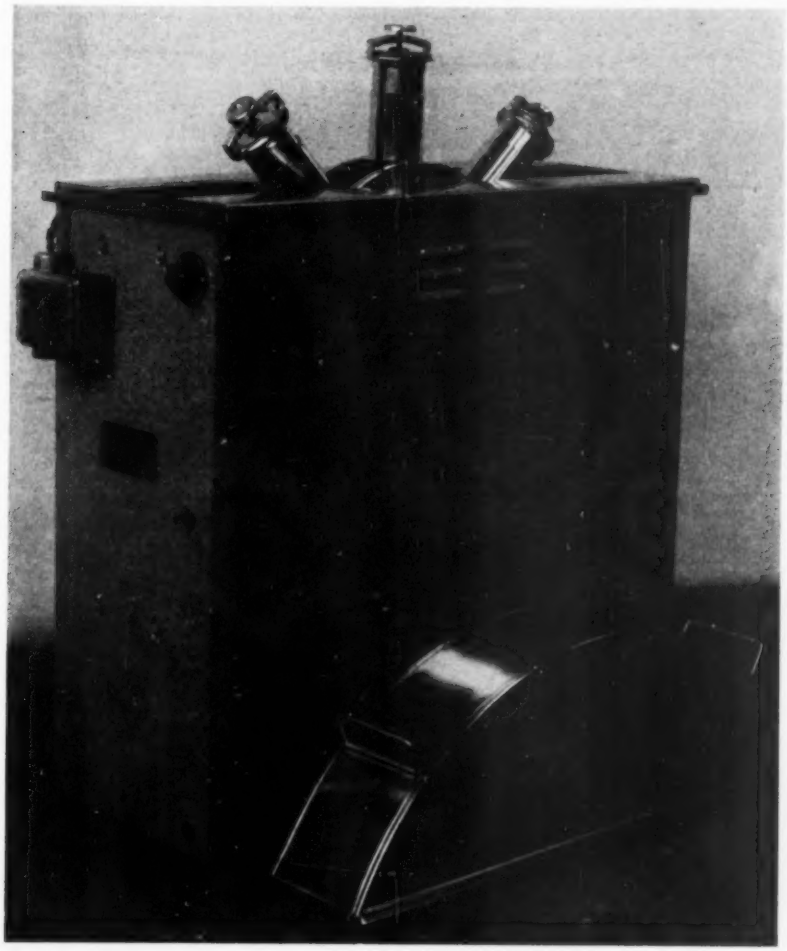

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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–10 of the January 1959 and pages 341–348 of the July 1959 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Solubility of Wool Dyes. Its Assessment and Practical Significance — *W. Beal*

The Remazol Linkage — *E. Bohnert*

Vat Dyeing of Cotton Piece Goods by Pigmentation Routes — *M. R. Fox and J. F. Mawson*

New Ideas in the Application of Phthalocyanine Derivatives to Textiles — *F. Gund*

Some Recent Advances in Textile Printing — *R. J. Hannay*

Tests for Colour Vision — *F. Jordinson and T. Minshall*

Aspects of Pigment Dispersion related to Usage — *I. S. Moll*

Some Relations between the Chemical Constitution of Cibacron Dyes and their Dyeing Characteristics — *J. Wegmann*

COMMUNICATIONS

The Dyeing of Cellulose Acetate with Disperse Dyes
VIII— Determination of Fibre Saturation Values — *C. L. Bird, H. K. Partovi, and G. Tabbron*

The Pad–Roll Dyeing System. A Summary of Experience — *R. Kern*

Comparison of a Colour Vision Test made from Dyed Wool Patterns with the Ishihara Test — *J. G. Wain*

How Much Research? A Critical Problem in the Manufacture of Textile Dyes — *G. S. J. White*

The Migration of Non-substantive Dyes on Fabrics and its Relation to Saturation Regain and Fibre Surface Structure — *D. Wilson*

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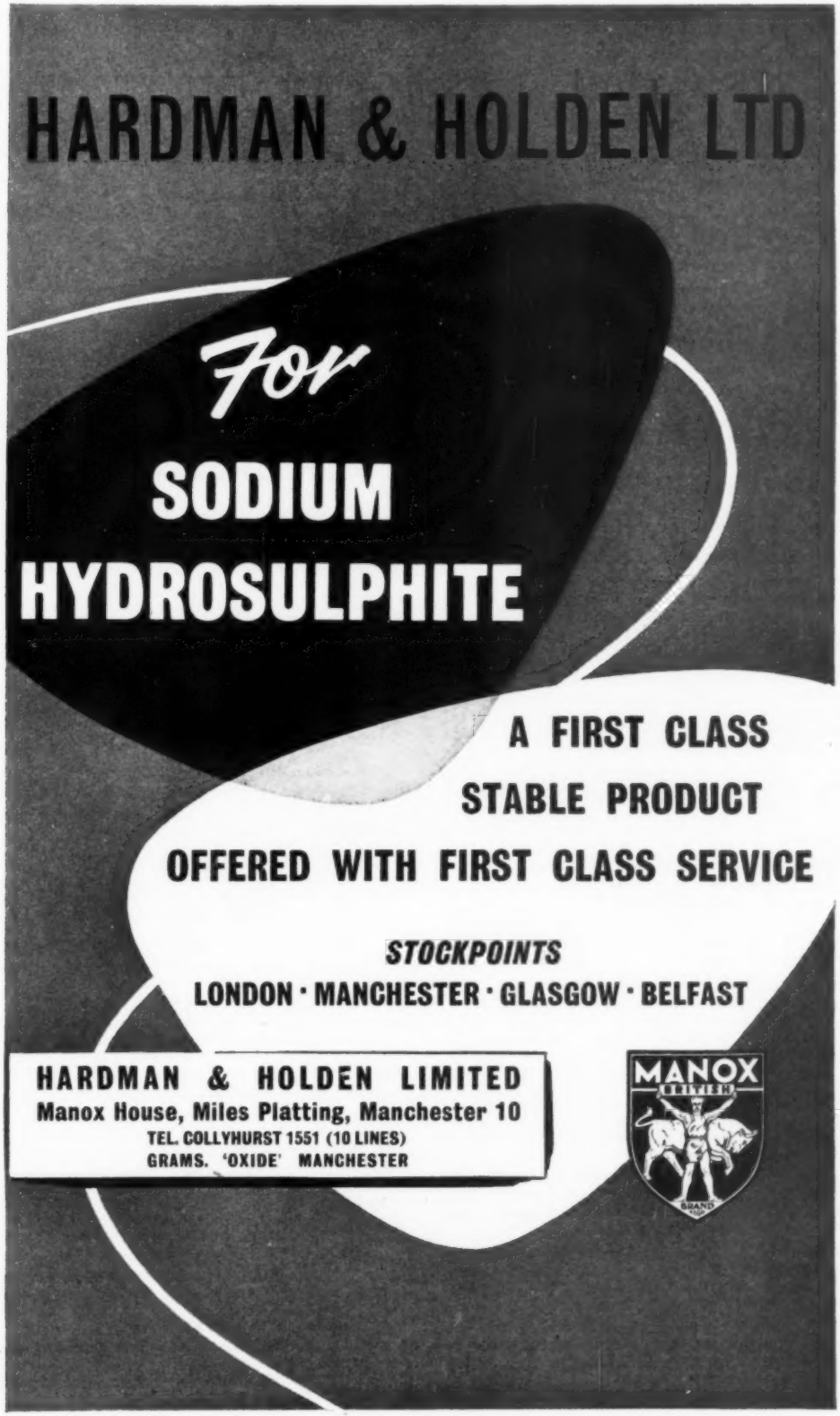
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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 15th December 1959

HUDDERSFIELD SECTION. *The Colouring of Tufted Carpet in Piece Form.* R. C. Cheetham Esq., G. W. Lewis, Esq. and Dr. J. Waller. (Courtaulds Ltd., Droylsden). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

SCOTTISH SECTION. *The Principles of Colour Photography.* Dr. C. H. Giles. Room 24, Royal College of Science and Technology, **Glasgow.** 7.15 p.m.

Wednesday, 16th December 1959

BRADFORD JUNIOR BRANCH. *Recent Developments in Wool Dyeing.* D. G. Evans, Esq. (CIBA Clayton Ltd.) Institute of Technology, **Bradford.** 7.15 p.m.

MIDLANDS SECTION. *Acrylics in Knitwear.* Half Day Symposium. J. F. Gaunt, Esq., Ph.D., F.R.I.C., F.T.I., H. D. Edwards, Esq., Ph.D., A.R.I.C., J. Rayment, Esq., A.T.I. College of Technology, **Loughborough.** 2.30 p.m. and 7 p.m.

Friday, 8th January 1960

LONDON SECTION. *New Works on the Dyeing of Nylon with Disperse Dyes.* H. W. Seaman, Esq., B.A. (I.C.I. Ltd.). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 12th January 1960

NORTHERN IRELAND SECTION. *Radio-isotopes and Their Industrial Applications.* R. J. Magee, Esq., M.Sc., Ph.D., M.R.S.A., A.R.I.C. (Queen's University, Belfast). Grosvenor Rooms, **Belfast.** 7.30 p.m.

SCOTTISH JUNIOR BRANCH. *Principles of Management.* W. A. Gray, Esq., B.Sc., A.R.C.S.T., A.M.B.I.M. (Senior Lecturer in Industrial Administration, Royal College of Science and Technology). Technical College, **Paisley.** 7.30 p.m.

Thursday, 14th January 1960

WEST RIDING SECTION. *Experience gained in the Dyeing and Finishing of Polyester/Wool Fabrics in Germany.* Dr. J. Nusslein (Farbwerke Hoechst). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

MANCHESTER JUNIOR BRANCH. *A Survey of Modern Textile Finishing.* G. Landells, Esq., B.Sc., Ph.D., A.R.I.C., F.S.D.C. Room J/E 11, Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 15th January 1960

MANCHESTER SECTION. *Wool the Modern Fibre— A review of recent developments in the Wool Research Field.* Dr. E. G. Carter (International Wool Secretariat). Room C.2, College of Science and Technology, **Manchester.** 7.0 p.m.

Monday, 18th January 1960

BRADFORD JUNIOR BRANCH. *Problems in the dyeing of Wool, Fibro and Terylene for blending.* Miss Esmée Smith (Wm. Smith & Co. Ltd.) Institute of Technology, **Bradford.** 7.15 p.m.

Tuesday, 19th January 1960

HUDDERSFIELD SECTION. *Problems in Dyeing Polyester Fibres and Means of Overcoming Them.* Dr. J. Nusslein (Farbwerke Hoechst A.G.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

LEEDS JUNIOR BRANCH. *Mechanism of Dyeing with Procion Dyes.* H. H. Sumner, Esq., B.Sc. (I.C.I. Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, the University, **Leeds 2.** 3.30 p.m.

MIDLANDS SECTION. *Beam Dyeing.* (Paper written by Burlington Corp. U.S.A.) K. S. Laurie, Esq. A.M.I.Mech.E., A.M.I.E.E. Gas Theatre, **Nottingham.** 7 p.m.

SCOTTISH SECTION. *Some Problems on the Dyeing and Finishing of Fabrics using the Newer Synthetics from the point of view of the customer.* J. S. Ingham, Esq., M.Sc., F.R.I.C. (Marks & Spencer Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Friday, 22nd January 1960

WEST RIDING SECTION. Annual Dinner and Dance.

Thursday, 28th January 1960

WEST RIDING SECTION. *Levelling Agents for Wool Dyeing.* Dr. J. Lanter (CIBA, Basle). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Friday, 29th January 1960

HUDDERSFIELD SECTION. Annual Dinner. Princess Cafe, **Huddersfield.**

Saturday, 30th January 1960

MANCHESTER SECTION. Annual Dinner and Dance. Grand Hotel, **Manchester.** 6 p.m. for 6.30 p.m.

Monday, 1st February 1960

HUDDERSFIELD SECTION. *Choice of an Artificial Daylight for Colour Matching with Special Reference to Fluorescent Materials.* F. L. Warburton, Esq., M.Sc., A.R.C.S., D.I.C., A.Inst.P. (Wool Industries Research Association). (Joint meeting with Huddersfield Textile Society.) Large Hall, College of Technology, **Huddersfield.** 7.30 p.m.

Tuesday, 2nd February 1960

LEEDS JUNIOR BRANCH. *Dyeing Anodised Aluminium.* A. E. Bratt, Esq. (CIBA Clayton Ltd.). The Lecture Theatre, Department of Colour Chemistry and Dyeing, the University, **Leeds 2.** 3.30 p.m.

NORTHERN IRELAND SECTION. *Professional Salesmanship— The Key to Prosperity.* A. B. M. Taylor, Esq. (Irish Linen Guild). (Joint meeting with Textile Institute.) York Street Flax Spinning Co. Ltd., Henry Street, **Belfast.** 7.30 p.m.

Friday, 5th February 1960

LONDON SECTION. *Dyeing and Finishing of 'Courtolon' Nylon in relation to its Yarn Properties.* L. Cotton, Esq., B.A., and J. Wardle, Esq. (Courtaulds Ltd.). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 9th February 1960

NORTHERN IRELAND SECTION. *Problems Relating to Permanent Finishes.* Dr. A. Schurch (CIBA Ltd., Basle). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Wednesday, 10th February 1960

MIDLANDS SECTION. *Dyeing and Finishing of Courtolon Yarns, Fabrics and Garments.* J. Wardle, Esq., L. Cotton, Esq., B.Sc. College of Technology, **Leicester.** 7 p.m.

Thursday, 11th February 1960

WEST RIDING SECTION. *Paper Chromatography of Dyestuffs.* J. C. Brown, Esq. (CIBA Clayton Ltd.). Griffin Hotel, Boar Lane, **Leeds 1.** 7.30 p.m.

MANCHESTER JUNIOR BRANCH. *The Photo-chemistry of Aromatic Molecules and Dyestuffs.* Professor G. Porter, M.A., Ph.D., F.R.I.C., Room J/E 11, Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

SCOTTISH SECTION *Recent Developments in Wool Dyeing.* W. Beal, Esq., B.Sc. (Geigy Co. Ltd.). (By invitation of the Textile Institute.) Scottish Woollen Technical College, **Galashiels.** 7.30 p.m.

SCOTTISH JUNIOR BRANCH. *Problems relating to Permanent Finishes.* Dr. A. Schurch (CIBA Clayton Ltd.). Technical College, George Street, **Paisley.** 7.30 p.m.

Friday, 12th February 1960

LONDON SECTION. Annual Dinner and Dance. Waldorf Hotel, **London W.C.2.** 7 p.m. for 7.30 p.m.

Tuesday, 16th February 1960

BRADFORD JUNIOR BRANCH. *Developments in the Manufacture and finishing of Terylene–Wool Fabrics.* J. B. Timis, Esq. (I.C.I. Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

continued on page xxxv

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COMMUNICATIONS

Studies on Haematoxylin and Haematein, the Colouring Principles of Logwood

I—Absorption Spectra of Pure Compounds in Various Solvents and a Spectrophotometric Method of Analysis for Haematoxylin and Haematein

G. C. Lalor and S. L. Martin

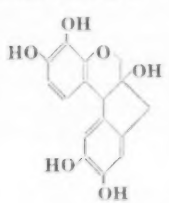
A modified method is described for the preparation from logwood extracts of pure haematoxylin. The criterion of purity is the ultraviolet absorption spectrum in methanol. In most solvents with low ultraviolet absorption, haematoxylin is either too insoluble or too susceptible to oxidation for suitable spectra to be obtained without special precautions.

The salient features of the visible and ultraviolet absorption spectra of haematein (about 10 mg. litre) in six solvents are given. In most of these haematein is eventually oxidised to give a product with a characteristically different spectrum. Solutions in dimethylformamide containing 1% of a phosphate–citric acid buffer are sufficiently stable for the haematein content to be determined by reference to the peak at 440 mμ. instead of the previously used peak at 280 mμ., which is shown to be unsuitable.

Exan ples of the application of the analytical method to commercial materials are given.

1. Introduction

Haematoxylin (I) and haematein (II), the colouring principles of logwood, are well known to be susceptible to oxidation, particularly in alkaline solutions [1]. The haematoxylin in logwood extracts is oxidised, during manufacture or in the dyebath, to haematein, which is the actual dyeing principle. There is little quantitative information in the literature on this oxidation step, the industrial control of which still rests largely on semi-empirical methods. Owing to the lack of satisfactory analytical methods, there is also uncertainty about the actual content of these two compounds in commercial extracts.



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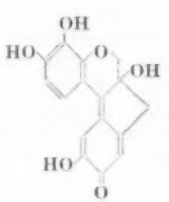

(II)

It was considered that kinetic studies on the oxidation step would be of value, but that before these could properly be undertaken further information was required on—

(i) A suitable criterion of purity for the two compounds
(ii) Their stability with time in solution
(iii) Methods for determining at least one in the presence of the other
(iv) Their behaviour in aqueous solution at varying pH, and their pK values.

The first three of these are considered in this paper, and the fourth in Part II, specifically using spectrophotometric methods.

There are very few accurate published data on the absorption spectra of either compound. Ringer [2] gave curves for both compounds, but did not specify his conditions sufficiently. The curve given by Teicher and Gordon [3] for their "haematoxylin reagent" obviously applies, from the results of our study, to material with haematein impurity. In reporting his curve for haematein in methanol, Inman [4] did not specify his experimental conditions, and the haematein may have been slightly oxidised in his solution. The absorption at 280 mμ. has been used [5] to determine the haematein content of certain commercial logwood products, but the full

* Published December 1959

spectrum was not given and, as shown in § 4, this choice of wavelength now appears questionable.

2. Experimental

2.1. MATERIALS

Haematoxylin

Freshly prepared liquid logwood extracts (made available by the West Indies Chemical Works Ltd. in Jamaica) were used. Repeated crystallisations from aqueous solutions using the previously reported methods[4–6] gave products the water of crystallisation in which depended on the procedure. The melting point could not be taken as a criterion of purity.

Good yields of needle-like crystals were obtained by crystallising below 15°c. from suitable extracts containing 1–2% sodium bisulphite to which small amounts of urea had been added; three recrystallisations from aqueous bisulphite solution in stoppered flasks at low temperature gave colourless crystals which were stored in mother liquor below 15°c. Before use, samples were filtered, washed with boiled-out distilled water containing a little sulphur dioxide, and placed in a vacuum desiccator below 15°c. This procedure lessened the risk of oxidation.

The criterion of purity eventually adopted was the absorption spectrum, with particular reference to the extinction coefficient at the maximum at 292 mμ., of solutions in methanol prepared under controlled conditions without heating and examined within 30 min. of preparation (after which the characteristics of haematein began to develop). Allowance was made for the water content, which was determined at the time of use with a quartz-spiral vacuum microbalance.

Haematein

Haematein was prepared by the peroxide oxidation of colourless haematoxylin[5]. The precipitate obtained on acidification was washed at the pump with glacial acetic acid, ether, ethanol, and water, and was converted to the crystalline form by heating an aqueous suspension on a water-bath for 30 min. The crystals were washed with ethanol and water, then dried over phosphorus pentoxide *in vacuo*; the water content was determined as for haematoxylin. The flat, red-brown crystals had a metallic lustre and decomposed at 240–250°c.

Solvents

All solvents were suitably purified by distillation, and checked for light-absorbing impurities before use.

2.2. APPARATUS AND PROCEDURE

Spectrophotometric measurements were carried out in an air-conditioned room at 25±1°c. with a Beckman D.U. ultraviolet spectrophotometer using 1-cm. matched silica cells with the requisite "blank" in the reference cell. Periodic checks were made of the wavelength calibration using the 334·1-mμ. mercury line, and of the photometer system using alkaline potassium chromate solutions.

The notation used is: k and E are respectively specific and molar extinction coefficients based on anhydrous solute; O.D. is optical density. Absorption curves are plotted against wavelength (mμ.), actual points being shown only where necessary to distinguish between curves.

3. Results and Discussion

3.1. HAEMATOXYLIN

Unheated methanol solutions were stable for more than 30 min. but eventually began to show absorption characteristics of haematein. Fig. 1A shows the absorption spectrum in methanol (36·9 mg./litre) of material recrystallised until a constant k-value at 292 mμ. was obtained with successive crystallisations. The absorption is negligible between 800 and 300 mμ., and slight between 320 and 310 mμ. (not shown); there is a sharp maximum at 292 mμ. with $k = 17{\cdot}04 \pm 0{\cdot}06$ ($E = 5{,}148$ for mol.wt. 302·3) and a sharp minimum at 256–257 mμ. ($k = 1{\cdot}58$ and $E = 780$).

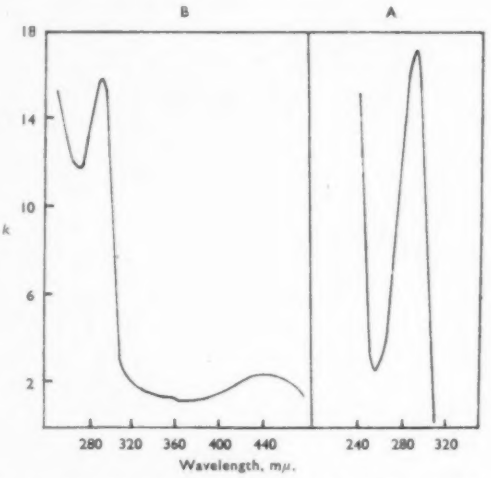

FIG. 1—Absorption Spectra in Methanol of (A) Pure Haematoxylin, (B) Dried Unoxidised Logwood Extract

For comparison, Fig. 1B shows the spectrum in methanol (42·4 mg./litre) of a vacuum-dried unoxidised logwood extract similar to Hematine Crystals NAD, and essentially a very crude haematoxylin containing a few per cent. of haematein; it can be clearly seen that the impurities lower the value of k at 292 mμ., increase that at 256 mμ., and cause absorption in the visible range.

It was difficult to obtain satisfactory spectra in solvents other than methanol and ethanol with low ultraviolet absorption, because haematoxylin was either too insoluble or too susceptible to oxidation in the solvents. In aqueous solutions above pH about 1·5 the haematein colour could develop rapidly; in other alcohols and in ether the behaviour was similar, although the rate of oxidation was lower.

3.2. HAEMATEIN

Haematein is completely insoluble in non-polar solvents, slightly soluble in water, alcohols, Cellosolve, dioxan, acetic acid, and acetone, and quite soluble in dimethylformamide (DMF).

Solutions of reasonable concentration could be obtained by first dissolving 1 mg. in 1 ml. DMF, then making up to 100 ml. with acetic acid, for example. Solutions in non-aqueous solvents, particularly acetic acid, dioxan, and DMF, were sufficiently stable for absorption measurements to be made. In DMF there were changes with time, apparently not due to oxidation, which were prevented by adding 1% of an aqueous MacIlvaine phosphate–citric acid buffer of pH 2·2. The solutions used contained about 10 mg./litre; a microbalance was used for weighings.

The salient features of the absorption curves for the solid and in different solvents are given in Table I, with a probable accuracy of 0·5% for the value of E_{max}. The data for the first specimen, with no solvent, were obtained using a fairly uniform layer of haematein on the outer face of the quartz cell (placed nearest the monochromator), formed by rapidly evaporating a saturated solution in methanol; an empty cell was used as the blank. The second column in Table I gives the approximate dielectric constant of the pure solvent in Debye units [7]. Fig. 2 shows log E as a function of wavelength for solutions in DMF with 1% phosphate–citric acid buffer and in acetic acid prepared as above. It is seen that there are in general at least two strongly characteristic peaks, one in the visible at 430–450 mμ., and the other in the ultraviolet region at 287–276 mμ., the actual wavelengths and E values depending on the

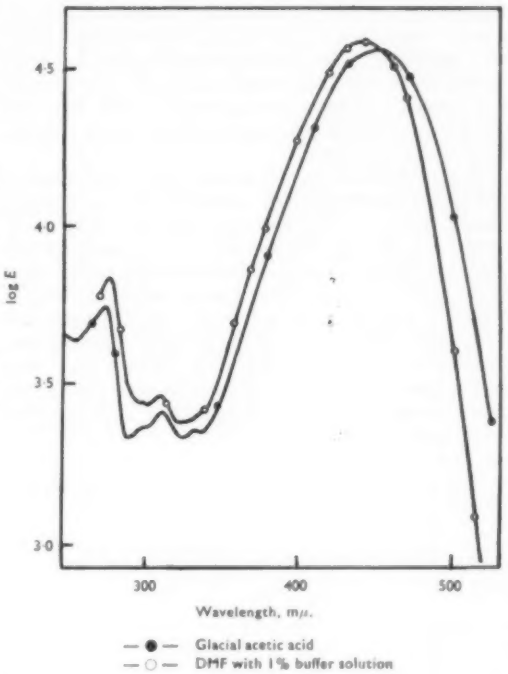

FIG. 2—Absorption of Haematein in Glacial Acetic Acid, and in DMF with 1% Buffer Solution

TABLE I

Salient Features for Haematein in Various Solvents at 25°C. (about 10 mg./litre)

Solvent	Dielectric Const. of Solvent	No. of Maxima	λ_{max}	E_{max}
None	—	3	450	—
			340	—
			286–287	—
Water (O_2 present, initial values)	78·5	4	558	Unstable with time
			445	
			288	
			276–278	
DMF (pure, somewhat unstable)	38	3	579	35,708
			442	30,124
			281	7,507
DMF + 1% buffer soln.	38 (pure)	4	440	39,250
			312	2,890
			296	2,940
			280	6,860
Methanol	32·6	4	445	35,490
			310	2,684
			295–297	2,946
			278	6,468
Acetone (visible region only)	20·7	2	576	2,045
			431·2	38,130
Acetic acid + 1% DMF	6·15 (pure)	4	450	36,720
			335	2,260
			313–314	2,573
			276	5,549
Dioxan	3	4	432	38,280
			310	2,762
			294–296	2,735
			278	6,726

solvent. It cannot be concluded from the present results that there is any consistent dependence on the dielectric constant of the solvent; it is to be noted, however, that the highest wavelength maximum (~578 mμ.) in acetone and in DMF, both containing a carbonyl group, is some 20 mμ. higher than in water.

With solutions in pure DMF, the maximum at 579 mμ. increased slowly with time (some 10% in about 3 hr.), while that at 442 mμ. decreased. Addition of 1% buffer solution suppressed the first peak and enhanced the second, which was shifted slightly to 440 mμ. and which no longer showed any marked changes with time. No attempts were made to investigate further the nature of the instability with time in the pure solvent or of the action of the added buffer (which may be associated with the known instability of DMF itself).

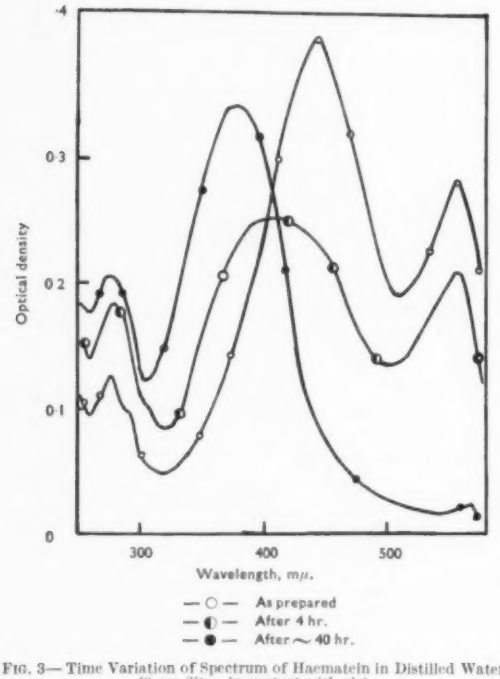

FIG. 3—Time Variation of Spectrum of Haematein in Distilled Water (9 mg./litre, in contact with air)

There were significant time changes with aqueous haematein solutions; therefore extinction values are not included in Table I. As shown by the curves in Fig. 3, these changes involved a gradual decrease in the absorption at 558 and 445 mμ., followed by a shift in the 445-mμ. maximum towards shorter wavelengths and almost complete disappearance of the 558-mμ. peak; concurrently, the ultraviolet absorption was gradually enhanced, the inflexion at 288 mμ. disappearing and the maximum increasing with a shift to longer wavelengths. Eventually the light yellow solution showed little of the haematein characteristics but rather those of a new compound with absorption maximum at 380 mμ. Curves of this type were obtained whenever haematein (or haematoxylin) in solution was completely oxidised under mild conditions (air, oxygen, hydrogen peroxide, sodium nitrite), and thus appear characteristic of unidentified oxidation products of haematein. Hematine Crystals ZA (WI) behaved similarly in aqueous solutions at equivalent concentration. Aqueous solutions of haematoxylin or of haematein prepared and stored in the complete absence of air did not show any noticeable changes over long periods of time.

The above results emphasised the need for special precautions in any spectrophotometric method of analysis, as shown in § 4 below, and in studies on these two compounds at varying pH, where oxygen has to be rigidly excluded, as shown in the subsequent paper (Part II).

4. Spectrophotometric Methods of Analysis of Haematein and Haematoxylin

4.1. HAEMATOXYLIN

The haematoxylin content of unoxidised logwood extracts can be readily obtained by applying the method of Morton and Stubbs [8] to the ultraviolet absorption spectra of methanol solutions. Thus, application of this method at wavelengths of 292, 298·8, and 284 mμ. to the data shown in Fig. 1B for a vacuum-dried unoxidised logwood extract indicates a haematoxylin content of 69%. The specific extinction coefficient of the haematein peak at 445 mμ. on this curve, with the E value at 445 mμ. for haematein in methanol given in Table I, gives a value of 2% for the haematein content of this material.

4.2. HAEMATEIN

Comparative dye testing on chrome-mordanted wool has always been the standard method for comparing haematein contents of commercial products. This method has certain advantages, but also some obvious disadvantages, particularly those related to the constancy of "standards", and to subjective judgment. Further, the method is of limited applicability to pure haematein (which is rather insoluble and easily decomposed under the dyebath conditions), and cannot be used for kinetic studies. Colorimetric methods have been described—a crude one [9] in 1885, and more recently methods using lakes with tin [10] or chromium [5]—but none has been generally used. Arshid *et al.*[5] also used the absorption at 280 mμ., recorded on a photographic plate from a spark discharge, of solutions (100 mg./litre in Cellosolve or methanol for pure haematein and in distilled water for Hematine Crystals) to determine the haematein content of logwood dyes, but did not report any special precautions for excluding the complications due to oxidation.

Primarily because of their better solubility and stability, it was decided to use solutions in DMF with 1% acid buffer. The wavelength of maximum absorption, 440 mμ., was chosen for analysis. Fig. 4 shows the Beer's-law plot over the range 2–11 mg./litre at a slit width of 0·06 mm., which was not, however, critical. The buffer concentration also was not critical, as with twice the normal amount the k value was reduced by only 1%. The k value with the normal amount of buffer was 130·7 $\pm$ 0·3.

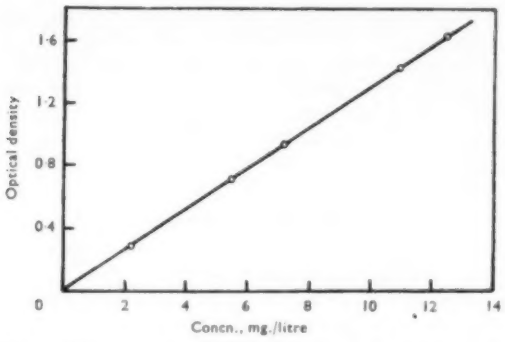

FIG. 4—Beer's-law Plot for Haematein in DMF with 1% Buffer Solution (slit width 0·06 mm.)

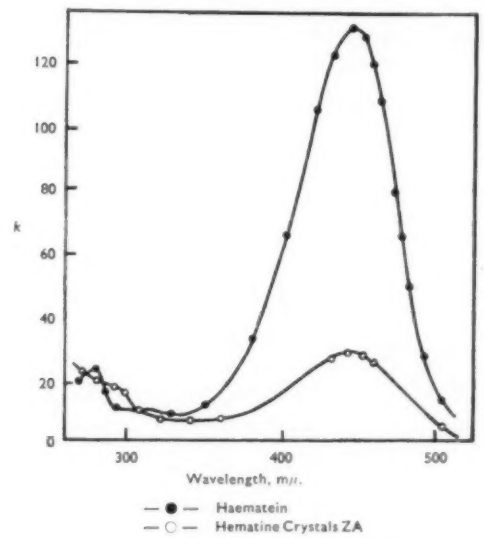

FIG. 5—Absorption Spectra of Haematein and Hematine Crystals ZA in DMF with 1% Buffer

Fig. 5 shows the absorption curves for haematein and Hematine Crystals ZA in the DMF–buffer solution (about 11 mg./litre). For the latter, the k value of 28·5 at 440 mμ. indicates a haematein content of 21·8%, compared with the value of 30% reported by Arshid *et al.*[5] for similar commercial materials using the wavelength 280 mμ. If the 280-mμ. maximum is used, the indicated haematein content is about 85%, an impossibly high figure, and the form of the curve in this region is quite different from that of pure haematein. The commercial products are usually considered to have tolerances estimated at only $\pm$ 5%. However, the results do suggest that in these products, known to contain large amounts of non-dyeing substances[5], the contents of non-dyeing material with strong absorption in the 280-mμ. region may vary widely. If correct, this would provide reasons additional to the oxidation effects for the unsuitability of the choice of this wavelength for analysis.

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Studies on Haematoxylin and Haematein, the Colouring Principles of Logwood

II—Behaviour in Aqueous Solutions at Varying pH, and the pK Values

G. C. LALOR and S. L. MARTIN

A method is described for measuring the absorption spectra of both compounds in aqueous solutions under conditions which minimise the risk of oxidation. Data so obtained are presented for concentrations 10^{-4} to 10^{-5}M. over the pH range up to 10. Despite the special precautions, oxidation effects are still observed with both compounds above pH 9–10. Below pH 6 haematein undergoes an acid-catalysed reaction.

There is no evidence of any ionisation process with haematoxylin solutions up to pH 9·6, above which reliable data could not be obtained. For haematein at 25°C. pK_a(I) is 6·86 $\pm$ 0·05 and apparently applies to the hydroxyl in the quinonoid ring, and pK_a(II) is 9·9 $\pm$ 0·1.

Absorption and kinetic studies suggest that in the acid reaction of haematein, catalysed by hydrogen ions in a pseudo-first-order manner, a molecule of water is added to form dihydrohaemateinol, by a reversible process proceeding to about 65% completion at equilibrium under the present conditions. The reaction is thus in some respects analogous to the reverse of the previously reported dehydration of hydroxy-di- or -tri-phenylmethanols to give the corresponding *p*-methylenequinones.

1. Introduction

In Part I the major difficulties in measuring the absorption spectra in aqueous solutions were shown to be the low solubility of haematoxylin in the cold and of haematein below pH 7, and the relative ease of chance oxidation of both compounds in aqueous solutions.

The solubility difficulties were overcome by first preparing solutions in small amounts of ethanol for haematoxylin and of dimethylformamide (DMF) for haematein, and then adding the requisite aqueous solutions. The oxidation effects were considerably reduced by using a simple apparatus to prepare solutions for absorption measurements with minimal contact with air or oxygen, and, where necessary, by obtaining zero-time extinction coefficients by suitable extrapolation.

Data on the effect of pH on the absorption spectra of aqueous solutions of both compounds are now reported, and estimates of the pK values are given for haematein.

It was hoped that, by working at low pH, where the oxidation of haematein itself is relatively slow, the kinetics of the oxidation of haematoxylin to haematein could have been studied. However, it was found that below pH 5 haematein undergoes another reaction at a rate dependent on pH. The kinetics of this reaction, studied in a preliminary manner, together with the absorption spectra data, suggest that it is an acid-catalysed reversible hydration of a type previously reported for certain other *p*-methylenequinones.

2. Experimental

2.1. MATERIALS

Both compounds were prepared and purified as in Part I. The aqueous systems used were: for pH < 2, 0·1 N. mineral acids; for pH > 8·5, 0·01 M-Na_2CO_3, 0·02 M-Na_2HPO_4 + 0·002 M-Na_2CO_3, and 0·04 M-Na_2HPO_4 solutions; for pH 2·2–8·5 MacIlvaine's buffers (varying proportions of 0·1 M. citric acid and 0·2 M-Na_2HPO_4 solutions) diluted five times to lower the total ionic strength.

2.2. PREPARATION OF SOLUTIONS

Solutions were prepared in the absence of air (vacuum from a rotary oil pump) in a simple apparatus[1]. By suitable manipulation of the various parts, the aqueous solvent, previously boiled out under vacuum, was added under vacuum to a weighed amount of haematoxylin or haematein— dissolved respectively in ethanol (6 mg. in 2 ml.) or DMF (1 mg. in 1·36 ml.)— to give a standard volume of 136·6 ml. After thorough shaking, a portion of the prepared solution was transferred under vacuum by further manipulation to an evacuated 10-mm. Beckman silica cell with a ground-in joint and stopcock, and the rest kept for pH measurements. With practice, the solution in the stoppered cell was ready for absorption measurements within 3–5 min. after the solvent was added. Checks showed that the solutions thus prepared were homogeneous.

2.3. MEASUREMENTS

Absorption measurements were made at 25 ± 1°C. as described in Part I, with a special cell-compartment cover to accommodate the stopcock attachment. For measurements specifically at 558 and 445 mμ. the slit-widths were 0·04 and 0·06 mm. respectively. Measurements of pH were made at room temperature (27–30°C.) on solutions prepared as above, except for those in 0·1 N. acids, with a Cambridge bench-type pH-meter standardised and checked with standard buffers.

3. Results and Discussion

3.1. EFFECT OF pH ON ABSORPTION SPECTRA OF HAEMATOXYLIN

The curves in Fig. 1 show no marked effect of pH up to 9·6, above which the haematein colour and characteristics developed too rapidly (in less than 3 min.) and the reproducibility was too poor for accurate measurements, despite the special precautions. There is no evidence of any ionisation process below pH 9·6, and above this data suitable for evaluation of pK values could not be obtained.

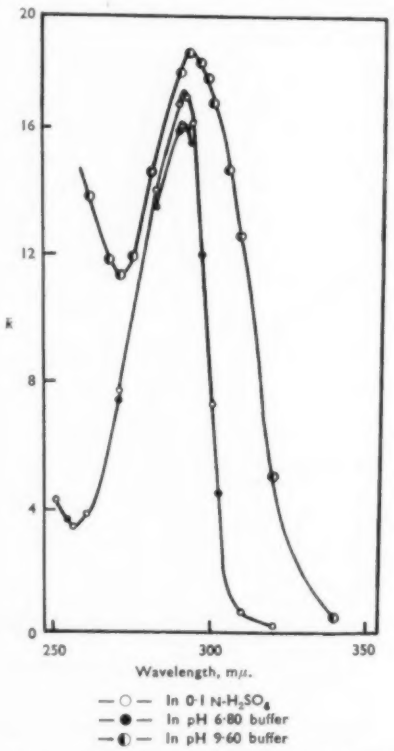


FIG. 1— Absorption Spectra of Haematoxylin Solutions (about 60 mg./litre)

3.2. EFFECT OF pH ON ABSORPTION SPECTRA OF HAEMATEIN SOLUTIONS

The stability with time was still too low at pH > 10 or < 5 for reliable results to be obtained for the entire spectrum. The time variation above pH 10 was typical of the oxidation described in Part I, indicating a limited value of the special precautions used here, as with haematoxylin. Below pH 5 the variation with time was quite different, as partly indicated in Fig. 3 and discussed fully in § 3.4.

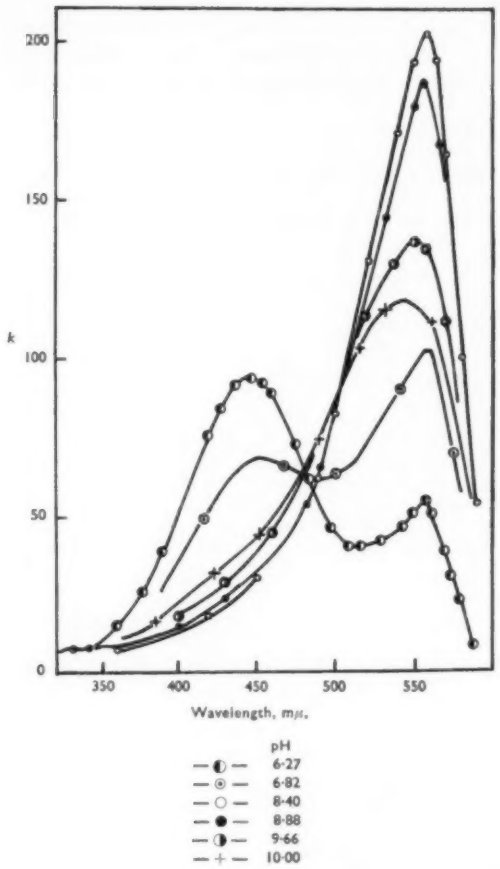


FIG. 2—Absorption Spectra of Haematein in Buffers at pH 6–10 (about 10 mg./litre)

Fig. 2 shows k as a function of λ for solutions in the pH range 6–10. Up to pH 8·4 a new band with peak at 558 mμ. gradually developed, while that with peak at 445 mμ. decreased. Above pH 8·4 this new band decreased, but the increase in the overall visible absorption between 520 and 380 mμ. suggested the appearance of a new band with head at 520 mμ.

In the ultraviolet, with decreasing pH below about 5 there was a suppression of the peak at 276 mμ. and development of a new peak at 290 mμ., but this was strongly time-dependent, as shown in Fig. 3, and is associated with the "acid reaction" discussed in § 3.4.

For solutions which showed marked time variations the procedure finally adopted was to measure the optical density at 558 and 445 mμ. as functions of time and extrapolate back to zero time to obtain zero-time extinction coefficients (k°).

3.3. pK VALUES FOR HAEMATEIN

The isosbestic point associated with the first ionisation process has not been located (Fig. 2 and 5), probably owing to the instability of solutions at pH below about 6, but that associated with the second process is clearly defined at 503 mμ. in Fig. 2.

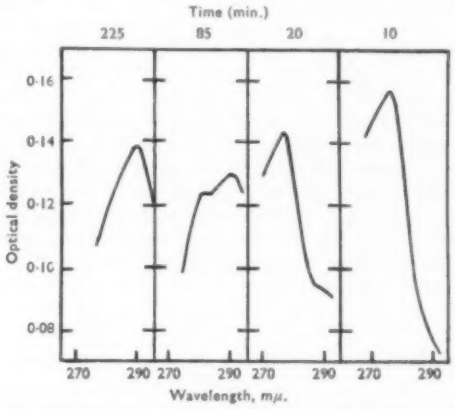


FIG. 3—Time Variation of the Ultraviolet Spectrum of Haematein at pH 4·30

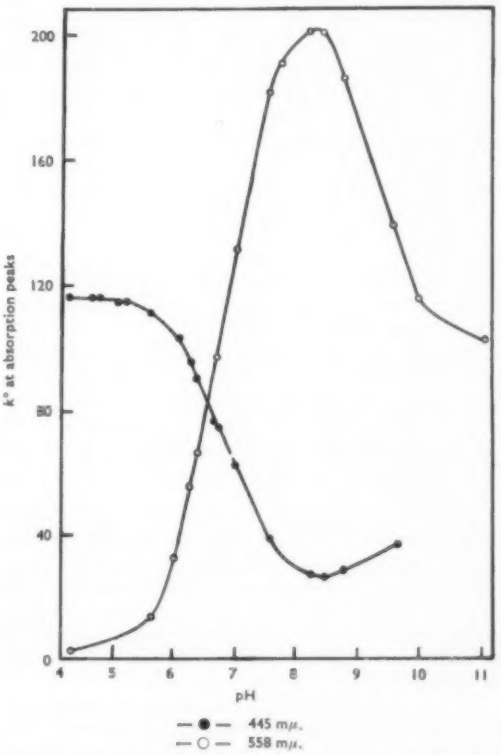


FIG. 4—Variation with pH of k° at 445 and 558 mμ. for Haematein Solutions

Values for k° at 558 and 445 mμ. were obtained from first-order plots of log (O.D.) against time for acid solutions (linear over at least the first 60% of the total change), and for other solutions (where the change was sufficiently slow) from plots of optical density against time which were linear over the first 10 min. These k° values are shown as

functions of pH in Fig. 4, from which the following k° values are indicated for the pure species of un-ionised (HA), singly ionised (A^-), and doubly ionised (A^{2-}) molecules—

	HA	A^-	A^{2-}
At 558 mμ.	1	203	102
At 445 mμ.	116	27	—

For the *first* ionisation process of a weak acid, assuming negligible difference in molecular weights of molecule and anion, and activity coefficients for molecule, hydrogen ion, and anion respectively of 1, 1, and f_{A^-}, the thermodynamic dissociation constant K_a can be obtained from the observed k° value at a particular pH by means of the following equation—

$$pK_a = pH - \log \frac{k^\circ(HA) - k^\circ}{k^\circ - k^\circ(A^-)} - \log f_{A^-} \qquad (i)$$

For the present solutions of weak ionic strength I, $-\log f_{A^-}$ is $0{\cdot}10 \pm 0{\cdot}01$ from the expression given by Biggs [2] with the I values calculated from published data [3]. Using the values for the species HA and A^- given in the table above with the observed k° values at the two wavelengths for four solutions in the pH range 6·27–7·60 (greatest stability), this equation gave as a mean of eight values $pK_a(I) = 6{\cdot}86 \pm 0{\cdot}05$. This agrees well with the value of 6·77 obtained by equating pK to the pH at the midpoints of the inflexions of the curves in Fig. 4, since the activity coefficient term is neglected in this case. The above choice of the extinction coefficients for the pure species HA and A^- was substantiated by the pK value of 6·79 obtained by the method of Rosenblatt [4], which obviates the need for such a choice by applying determinants to three equations involving observed k° values at three different pH values, and neglects the activity coefficient term.

For the *second* ionisation process, the requisite expression [2] for $-\log f_{A^{2-}}$ in the equation corresponding to (i) with the observed k° at 558 mμ. for pH 9·6 gave $pK_a(II) = 9{\cdot}9 \pm 0{\cdot}1$, compared with the value of 9·4 obtained by equating pK to the pH at the midpoint of the second inflexion on the curve for 558 mμ. in Fig. 4.

Arshid *et al.*[5] obtained $pK(I)$ 6·5 and $pK(II)$ 10·3 from potentiometric titration curves with caustic potash, in the presence of nitrogen, on haematein (400 mg./litre) in boiled-out distilled water plus 10% Cellosolve, but the points of inflexion on their curve were not very clearly defined and their precautions against oxidation might not have been as effective as in the present work.

The pK values for simple substituted phenols or polyphenols such as catechol [6] range from 9 to 10, suggesting that the first ionisation process in haematein, with pK 6·86, must involve the hydroxyl *ortho* to the quinone group. The second process observed here must then involve one or both of the phenolic hydroxyls, since the present method did not permit differentiation between a second and a third process.

3.4. BEHAVIOUR OF HAEMATEIN IN ACID SOLUTIONS

As already mentioned, the absorption spectra indicated anomalous behaviour below pH about 6. The more important characteristics were as follows—

(i) The reproducibility was poor, and apparently time-dependent to an extent which increased as the pH decreased.

(ii) At 445 mμ. k°(HA) was 116, but the observed k values in 0·1 N. strong acids (no ionisation expected) were consistently 12–14 as shown in Fig. 5, were only slightly higher in 0·1 N. weak acids, and appeared to depend on pH.

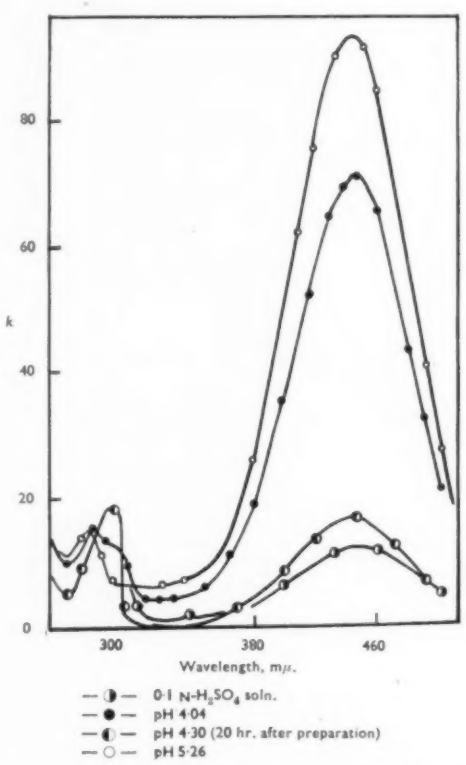


FIG. 5—Absorption Spectra of Haematein in Acid Solutions

(iii) The ultraviolet characteristics were markedly altered in a time-dependent manner different from that associated with oxidation (Fig. 3; also cf. curves in Fig. 5 with that for pH 8·88 in Fig. 2). A peak at 290 mμ. replaced that at 276 mμ.

(iv) The "fading" of the visible colour at low pH was also different from that due to oxidation, was dependent on pH, and did not occur in glacial acetic acid (Part I), although it did occur in 0·1 N. acetic acid solution.

(v) The haematein content apparently never disappeared completely, and the rate of fading decreased very rapidly after some 70% of the change had occurred, suggesting an equilibrium reaction.

These observations suggested the occurrence of a previously unreported acid-catalysed equilibrium reaction of haematein involving water and giving a product with ultraviolet absorption similar to

that of haematoxylin, i.e. in which the conjugation in the haematein molecule had been disrupted.

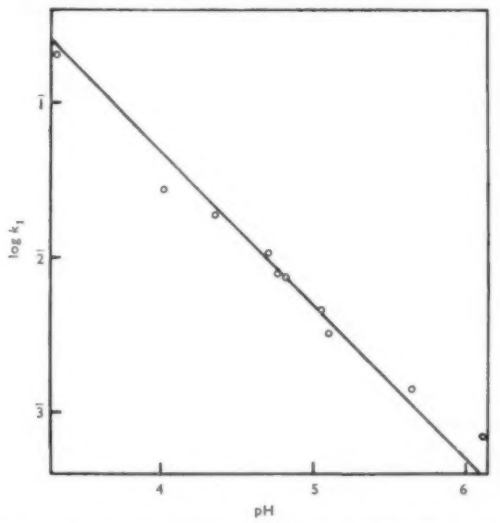
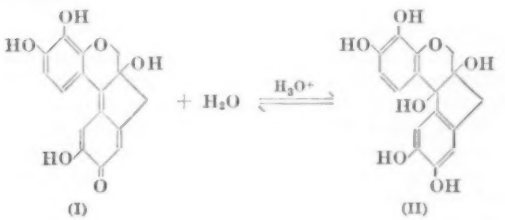


FIG. 6— Log (velocity constant k_1) for Acid Reaction of Haematein as a Function of pH

Kinetic studies showed the change at 445 mμ. to be first order with respect to haematein over some 60% of the reaction. Reaction velocity constants k_1 (obtained from the slopes of the linear plots of log (O.D.) against time) were directly proportional to the hydrogen ion concentration, as shown in Fig. 6, where log k_1 plotted against pH over the range 3–6 has negative unit slope. (Below pH 3 the reaction is too fast and above pH 6 the spread of points too wide for accurate data to be obtained by the methods used.) The reaction is thus pseudo-first order with respect to hydrogen ion concentration.

The above evidence suggests the following reaction—



(I) (II)

The pentamethyl derivative of compound II, dihydrohaemateinol, was prepared by Perkin *et al.*[7]. The brazilein analogue of II has been postulated by Morsingh and Robinson[8,9] as an intermediary formed on hydrolysis of the acyl compound with sodium hydroxide, which on acidification loses water to form DL-brazilein. Robinson[9] states that haematein can similarly be prepared from compound II, i.e. by the reverse of the above reaction. There are also many recorded instances[10] of hydroxydiphenylmethanols and hydroxytriphenylmethanols being converted to *p*-methylquinones by loss of water, essentially the reverse of the above reaction; many of these conversions are reversible, and some of the carbinols (analogues of compound II), particularly in the aurin and benzaurin series, cannot be obtained in the free state.

* * *

The authors wish to thank Professor C. H. Hassall and Mr. E. L. F. Mucklow, initial discussion with whom suggested the work described in these two papers; Mr. David Spoerri and the West Indies Chemical Works Ltd. for the provision of raw materials and other facilities and for their general goodwill; and Professor L. J. Haynes for helpful discussion on the acid reaction described in Part II.

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Optical Studies

I—Relation of Colour to the Apparent Soilability of Textile Materials

H. B. MANN and T. H. MORTON

The apparent degree of soiling of a coloured object is a matter of subjective assessment, but is, as we have been able to show with a group of observers, closely paralleled by the physical quantity H_s, the diminution (%) in the brightness coefficient of the surface—

$$H_s = \frac{Y_0 - Y_s}{Y_0} 100$$

A study of soiled coloured starch has indicated the possibility of relating H_s and hence degree of apparent soiling to the physical concentration of soil and the reflectance spectrum of the original surface. It is possible to compute from the spectrum a characteristic empirical quantity, the SMR value, which ranks the coloured material in order of relative apparent unsoilability in a series of materials, similar in structure but differing in colour. The derivation of SMR values for coloured starch involves no optical hypothesis, and the generalisation to fibrous materials depends only on the essential optical similarities of the systems. Full practical details, with tabulated data, are given for the determination of the SMR values of coloured fibres. The conclusions are discussed with special reference to the behaviour of carpets made from viscose rayon staple.

1. Introduction

The rapid increase, during the last four years, in the use of viscose rayon staple as a surface fibre in carpets brought with it urgent problems, now happily resolved, concerned with soiling. In part these were due to faulty manufactures, in which oily impurities left in the pile increased considerably the rate of physical accretion of black soil. Such manufactures were quickly corrected by appropriate methods discussed elsewhere in the technical literature. In part, however, the problems were concerned with the colour of the carpet pile, and it was found that full white, pastel colours, and bright yellows, of a type not generally attainable on carpet wools, gave rise to a series of consumer complaints of rapid and unacceptable soiling.

The present work was undertaken three years ago as a measure to meet this situation. We have been able to classify the relative unsoilability of a coloured carpet fibre by means of a quantity, the SMR value, computed from its spectral reflectance curve. For more than two years the SMR-value method has been widely applied to carpet dyeings and has proved, as hoped, an excellent guide to action.

In the work described below we have detailed our reasoned approach to the problem, and have indicated the solution stage by stage. The definition of SMR value, we believe, breaks new ground in textile optics: while it was primarily introduced for use with carpet dyeings, it has been found to be useful in a variety of circumstances concerned with the soiling of apparel and furnishing textiles, and even in co-ordinating the observed dependence of opacity of an apparel fabric on its coloration.

2. General Approach

2.1. SOILING OF TEXTILES

All textile materials soil in use. Clothing textiles are washed or dry-cleaned as often as is necessary to keep them in a fresh state. It is notable here that men's suits, which are relatively infrequently cleaned, are normally coloured relatively dark or drab. Conversely, washable garments are generally produced in white or light colours. Work clothes for dirty occupations are almost always coloured dark blue, bottle green, or a deep brown. There is thus *a priori* evidence that the colour of the textile article is an important factor in determining its apparent rate of soiling.

Certain textiles, notably permanent upholstery and carpets, present a special aspect of the problem, since cleaning is done, if at all, at very long intervals. Carpets represent the extreme case, since infrequency of cleaning is combined with use under extreme conditions of soiling.

2.2. NATURE OF SOILING

Soiling may be defined for present purposes as a deposition of dark-coloured foreign matter on the textile fibres in such a way that the soil is not readily removed by mechanical means; this soil consists of greasy matter together with dark-coloured sooty particles of microscopic or sub-microscopic size adhering firmly to the surface of the textile filaments. There is also general loose débris, which can be removed, for example, by shaking, brushing, or the use of a vacuum cleaner. In the case of carpets the amount of removable débris can be very great (we have, on occasion, found 1 lb. of dirt per square yard removable by vacuum cleaning), but even the presence of such large quantities of removable material does not alter significantly the appearance of the textile material. The cause of this is obvious under the microscope: the change in appearance is due to the adhesion of very small dark-coloured particles, probably sooty, such as are present in all urban and most country atmospheres. This particulate soil is attached firmly to the surface of the fibre, very often on special sites such as the striations in viscose rayon or the scale margins in wool. Because of its particulate nature, soil is confined to the surface of the filament: there is never any penetration of the fibre substance, such as is normal with dyes.

Soil particles under the microscope are black or nearly so. Some we have seen are a very dark brown, rather than a full black; but it may be taken that these are a variant of the general case, which comprises soiling with particles opaque to the

whole of the visible spectrum. For this reason we believe that a Carbon Black pigment (C.I. Pigment Black 7) may be used as a model soil in the optical sense.

2.3. STATEMENT OF OPTICAL PROBLEM

We have sought to discover the relationship between the colour of the material soiled and the apparent or visible change due to application of soil. This is partly an optical problem, but it contains also an element of subjective estimation depending on the structure of the eye and the nature of sensory perception mechanisms.

A full solution of the problem involves four stages—

(*a*) Soiling causes a change in appearance. The physical difference is a change in the amount and the quality of the light reflected from the soiled textile, but no objective argument can establish which physical quantity corresponds to the difference in sensory perception. It is necessary, therefore, to ask a group of observers to arrange a number of pairs of unsoiled and soiled materials of various colours in order of apparent soiling, and then to find the physical quantity giving the same ranking. This is the essential step in correlating subjective assessment with optical measurements.

(*b*) It is then required to relate the change in the relevant physical quantity to the amount of soil such as Carbon Black pigment present in the textile material.

(*c*) The optical investigation must be of such a nature as to permit calculation of the experimentally observed change in the relevant quantity from the spectral absorption curve of the unsoiled fibre, and the known content of soil.

(*d*) Finally the calculated change in appearance must be expressed in a form which indicates the relative rate of apparent soiling of the sample under consideration (compared with a white sample).

2.4. STARCH AS AN OPTICAL MODEL

All textile fibres are more or less transparent rods of small diameter; they have a large specific surface area on which soil particles can become attached.

There are serious, and at present insurmountable, experimental difficulties in making quantitative soiling experiments on textile fibres. A model substance such as starch presents a number of experimental advantages. General considerations indicate that it is an optical model differing not too widely from textile fibres; its main difference is that it is divided into granules rather than into rodlets as in the case of textile fibres. Corn starch appears to be the best available material, and its advantages may be listed—

(*a*) Uniform small grain size, ca. 15 μ. in diameter

(*b*) Grains are transparent and of very low light absorption

(*c*) Specific surface area is of the same order as for a textile fibre

(*d*) Coloration to any required shade is possible by a wide selection of dyes

(*e*) Uniform soilability with Carbon Black pigment by a milling technique.

With this model, therefore, it is possible to avoid the experimental difficulties encountered in the soiling of textile fibres and at the same time to study the interaction of dye and soil in the absorption of light in a structure not very dissimilar from that of a compact textile material.

3. Apparent Soiling of Dyed Starch Samples

3.1. COLOURS INVESTIGATED

A series of eight colours were dyed on corn starch by methods given in detail in § 6. It was intended that the dyed samples should give a rough coverage of the whole colour solid. Five of the samples were rough matches to carpet-fibre colours, and three other colours and the original white were added to give type coverage.

TABLE I

Colours of Dyed Starch Samples

Sample No.	Colour	*B.S.* 543:1934 BCC	Dyes	*Colour Index* Designation
1	White (undyed)	1	—	—
2	Lemon	52	Solophenyl Yellow GFL	C.I. Direct Yellow 50
3	Crushed strawberry	158	Benzanil Red BLL	—
4	Claret	36	Direct Fast Scarlet SE	C.I. Direct Red 23
5	Saxe blue	45	Chloramine Sky Blue FF	C.I. Direct Blue 1
6	Almond green	10	Direct Green 2G	C.I. Direct Green 8
7	Silver grey	153	Durazol Blue 2R Solophenyl Brown GL Solar Orange RGL	C.I. Direct Blue 71 C.I. Direct Brown 115 C.I. Direct Orange 62
8	Between mineral blue and victrix blue	46–47	Durazol Blue 2R Formal Black 2B	C.I. Direct Blue 71 —
9	Terra cotta	133	Durazol Brown 8R Solophenyl Brown GL	C.I. Direct Brown 112 C.I. Direct Brown 115

The colours are named in Table I, where the nearest colour in the British Colour Council *Dictionary of Colour Standards* (*B.S.* 543:1934) is given.

3.2. SOILED SERIES

Each of the samples 1–9 in Table I was soiled by milling with varying amounts of a standard Carbon Black pigment—2, 5, 10, 20, and 50 parts of added pigment per 10,000 parts of starch. The soiled samples may be conveniently coded—

Samples 1_0, 2_0, 3_0, ... are unsoiled samples
1_2, 2_2, 3_2, ... are samples soiled with 2/10,000 Carbon Black

etc.

Reference has already been made in § 2 to the difficulty of dispersing soil particles uniformly. It was found with the starch samples that reproducibility in reflectivity was attained, but only after milling for many hours at every stage of the preparation of the soiled samples. Full details of the necessary practical operations are given in Appendix I (§ 6). A preliminary examination of the series indicated that white showed soiling very much more than any of the dyed samples. Of the dyed colours the yellow (Sample 2) was highest in apparent soiling. At the other end of the scale there was no doubt that the blue (Sample 8) showed less apparent soiling than any of the other series.

3.3. RANKING OF APPARENT SOILING

It was found convenient to observe each starch sample in a glass sample tube of $\frac{5}{8}$ in. diameter. It was decided, after the rejection of a number of possible alternative schemes, to ask a group of 20 observers to rank the nine pairs $n_0 : n_{20}$ (n = 1–9) in descending order of apparent soiling.

The results of the ranking by the observer group are given fully in Table II. The mean ranking was obtained by adding each file across and dividing the sum by the number of observers. By setting the mean rankings in order, the mean order of the whole group is obtained; this is recorded in the last column of the table.

The correlation of individual observers with the mean order is very high, so that it is evident that all observers are ranking the samples for the same property. In other words, there is apparently unanimity within the group of 20 people examined as to the subjective entity defined by the term "soiling". A quantitative measure of the correlation of each observer is given by Spearman's rank-correlation coefficient—

$$\gamma = 1 - \frac{6\Sigma\Delta^2}{n^3 - n} \qquad \text{(i)}$$

where n is the number of objects ranked and Δ is the difference between observed and mean order for a given sample.

The rank-correlation coefficient is 1·00 for perfect agreement and zero for no correlation. For a group of nine objects the numerical value of the coefficient can be interpreted thus—

Coefficient 0·7 ... Correlation probably significant (5% probability level)
0·9 ... Correlation highly significant (0·1% probability level)

TABLE II

Ranking in Decreasing Order of Apparent Soiling of the Series s = 20

Sample Pair	Order by Observer																				Mean Ranking	Mean Order	Mean Ranking, Observers 1–11
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20			
1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1·00	1	1·00
2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2·00	2	2·00
3	6	8	6	6	8	6	6	7	7	7	7	8·5	6	5	5	5	4	7	5	6	6·33	7	6·82
4	5	5	5	3	3	3	3	4	3	3	3	3	4	3	3	3	3	6	3	3	3·45	3	3·45
5	8	8	6	8	6	8	8	8	8	8	8	6	5	8	8	8	6	8	8	8	7·45	8	7·63
6	4	4	5	5	5	5	5	6	5	6	5	4	3	4	6	7	7	4	8	7	5·25	5	4·91
7	3	3	4	4	4	4	4	3	4	4	6	5	8	7	4	6	5	3	4	5	4·50	4	3·91
8	9	9	9	9	9	9	9	9	9	9	9	8·5	8	9	9	9	9	9	8	9	8·88	9	9·00
9	7	6	7	7	7	7	7	5	6	5	4	7	8	6	7	4	8	5	6	4	6·15	6	6·27
γ	0·95	0·96	0·96	0·99	0·96	0·99	0·99	0·98	1·00	0·99	0·95	0·94	0·78	0·91	0·96	0·90	0·87	0·93	0·91	0·94	1·00	—	1·00

For a variety of reasons, observers 1–11 were considered most reliable. A separate analysis of this group gives a mean order identical with that for the whole 20 observers. Very great confidence can therefore be placed in the recorded mean order as a precise guide to the relative apparent soiling of the nine starch samples examined.

3.4. NOTE ON SOME OBSERVERS

Three observers had had considerable practical experience of colour matching. It is interesting that none of the three is in the group of the 11 best observers. We believe here that the colour matchers, although consciously trying to discount the fact, may have been subconsciously equating soiling to their knowledge of the amount of flattening dye required to produce the change in colour.

Two of the observers are known to have abnormal colour vision—

Observer 2 is a protanomalous trichromat, to whom red is of somewhat lower brightness than to a normal observer. His greatest divergence from the mean order is in setting the bright red (Sample 4) two places down in the ranking.

Observer 15 has a more serious abnormality: he is a dichromatic deuteranope. A characteristic of this type of abnormality is difficulty in certain colour discriminations, but with a perception of brightness approximating to that of a normal observer. His correlation with the mean is extraordinarily good; the significance of this will become apparent when the results of § 4 are taken into account. It was, however, felt that this observer should not be included in the list of the 11 best observers.

4. Optical Measurements and Derived Quantities

4.1. REFLECTANCE MEASUREMENTS

Light reflected from the various samples was conveniently measured with the EEL photoelectric reflectometer by methods described in detail in Appendix II (§ 7), where the numerical data will be found in Tables VII–IX.

4.2. Y AND H VALUES

The Y value of the sample is the optical function expressing the relative brightness of a surface as seen by a normal eye. It has been defined by the C.I.E. as a quantity derivable from the spectral reflectance of the sample. In the present paper it is expressed as the percentage of the brightness of a magnesium carbonate surface (considered to be 100%). The EEL photometer gives an approximate Y value for tungsten light from a single reading using a special filter.

The relative loss of brightness (%) after soiling is given by the equation—

$$H_s = \frac{Y_0 - Y_s}{Y_0} 100 \qquad \text{(ii)}$$

where Y_0 is the brightness of the unsoiled sample and Y_s is the brightness after soiling with a concentration of s parts of Carbon Black per 10,000 parts of starch.

TABLE III

Brightness Coefficients and Derived Quantities

Sample	Experimental			Calculated		
	Y_0	Y_{20}	H_{20}	Y_0	Y_{20}	H_{20}
1	96·7	29·9	69·0	96·5	29·8	69·1
2	51·5	24·3	52·8	50·6	25·2	50·1
3	12·2	9·4	23·0	14·7	10·4	29·3
4	18·0	11·8	34·4	20·5	13·0	36·6
5	11·0	9·1	17·3	12·3	9·7	21·2
6	22·0	16·1	26·8	22·8	15·7	31·0
7	37·2	25·7	31·0	37·4	21·6	42·3
8	8·4	7·4	11·9	9·0	7·6	15·6
9	19·0	14·0	26·3	20·3	14·0	31·0

In Table III are listed the experimental values for Y_0, Y_{20}, and H_{20} for the nine samples used in establishing the relative subjective soiling order (§ 3).

TABLE IV

Ranking of Brightness Coefficients compared with Ranking for Apparent Soiling

Sample	Order of Ranking					
	Experimental			Calculated		Subjective Mean Order
	Y_0	Y_{20}	H_{20}	Y_0	H_{20}	
1	1	1	1	1	1	1
2	2	3	2	2	2	2
3	7	7	7	7	7	7
4	6	6	3	5	4	3
5	8	8	8	8	8	8
6	4	4	5	4	5·5	5
7	3	2	4	3	3	4
8	9	9	9	9	9	9
9	5	5	6	6	5·5	6
γ	0·93	0·90	1·00	0·95	0·98	—

Table IV gives the ranking of these quantities in comparison with the mean order of soiling found by the observer group. It will be noted that H_{20} (experimental) correlates perfectly with the subjective soiling order. A relative loss in surface brightness may therefore be equated to apparent soiling: it is the required measurable physical quantity expressing the extent of visible soiling. The first stage in the soiling problem as defined in § 2.3 has therefore been solved.

The brightness of an unsoiled sample is highly, but not perfectly, correlated with apparent soilability of colour. This implies that in general dark colours will show soiling least. There are, however, notable exceptions: for example, the claret (Sample 4), of low brightness but high saturation, shows a relatively high tendency to soil. It is concluded that the brightness of a sample is a rough but not absolute guide to its soilability.

The brightness of the soiled samples Y_{20} is less highly correlated with the subjective soiling order. This correlation is of little importance for our present purpose.

4.3. TREATMENT OF DATA BY MEANS OF THE N FUNCTION OF R

It has been found convenient to define a function—

$$N = \frac{1 - R^2}{2R} \qquad \text{(iii)}$$

where R is the *fractional* monochromatic reflectance. It should be noted that there is no

theoretical basis for this function. It is used as an empirical but convenient function for further computation.

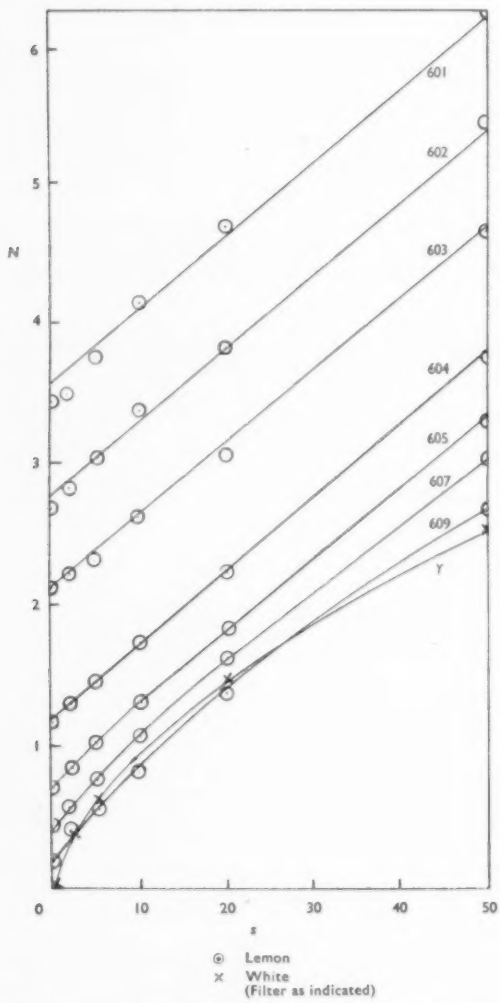

FIG. 1—*N*-Function *versus* Soil Concentration for White and Lemon

In Fig. 1 N is plotted against s for the undyed sample (R is derived from the Y value) and for monochromatic reflectance of the lemon (Sample 2) at a number of wavelengths covering the whole visible spectrum. This plot has a number of advantages—

(*a*) The white curve approximates to—

$$N_s = as^{\frac{1}{2}} \qquad \text{(iv)}$$

(*b*) The upper curves are parallel and approximate to—

$$N_s = N_0 + bs \qquad \text{(v)}$$

(*c*) Intermediate curves are of mixed type—

$$N_s = N_0 + axs^{\frac{1}{2}} + b(1 - x)s \qquad \text{(vi)}$$

The approximate value of x in (vi) becomes smaller as the initial point of the curve N_0 becomes higher. It has been found empirically that the value of x can be represented as an exponential function of N_0. The equation—

$$N_s = N_0 + 0{\cdot}35(10^{-1{\cdot}67N_0)}s^{\frac{1}{2}} + 0{\cdot}052\,(1 - 10^{-1{\cdot}67N_0)}s \qquad \text{(vii)}$$

fits the data of Fig. 1 within the experimental error.

If N_0 is set at 0·031, the soiling equation for *undyed* starch becomes—

$$N_s = 0{\cdot}031 + 0{\cdot}311s^{\frac{1}{2}} + 0{\cdot}00585s \qquad \text{(viii)}$$

On the basis of this result it may be expected that (vii) may be used to express the reflectance of any white soiled sample or the monochromatic reflectance of any coloured sample soiled or unsoiled. The relationship is such as is required to fulfil the condition set out in § 2.3 (*c*).

It will be noted that in Fig. 1 the white curve cuts and crosses the reflectance curve of the lemon sample reflecting monochromatic far red light. This crossing is beyond experimental error and is probably due to a slight change in granule adhesion after dyeing from an alcohol solution, thus altering the scattering factor. This crossing of the curves for white and the coloured curves at high reflectance is implicit in the empirical equation (vii). Although it represents a physical impossibility, we thought it better to retain a fit with the experimental data, especially as the small degree of crossing appears to introduce no significant error into the soiled reflections as finally computed.

4.4. CALCULATED SPECTRAL REFLECTANCE OF SOILED SAMPLES

Equation (vii) can now be used to set up tables of expected monochromatic reflectance for particular degrees of soil. We have calculated the expected monochromatic reflectance at $s = 20$ for R ranging from 5 to 100 by steps of 1 (Table X, Appendix III). The relationship of R_{20} to R_0 is shown graphically in Fig. 2.

The effect of the crossing of curves referred to in § 4.3 is shown by the slight maximum in the plot of soiled reflectance against initial reflectance: had there been no crossing, the curve would have risen from zero at lower values of R_0, attaining a flat maximum at $R_0 = 0{\cdot}70$–$1{\cdot}00$. It is interesting to note that the reflectance of such soiled samples is virtually constant at approx. 30% for all values of the original monochromatic reflectance between 65% and 100%.

4.5. AGREEMENT OF CALCULATED AND OBSERVED REFLECTANCES

Equations (vii) and (viii) may be used to predict the reflectances of soiled samples; their effectiveness can be shown by plotting experimental against calculated data as in Fig. 3–5. Fig. 3 plots the expected reflectances of undyed starch over the whole range of soiling. Fig. 4 gives the experimental and calculated reflectances for the lemon yellow (Colour 2) soiled at $s = 20$ for the whole range of the visible spectrum; Fig. 5 gives similar reflectances for claret (Colour 4).

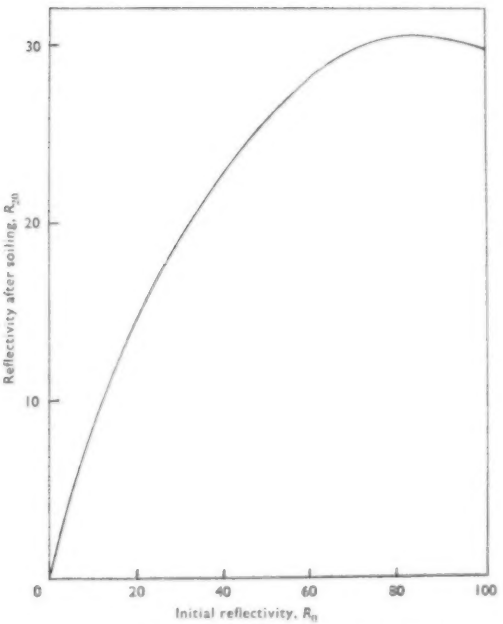

FIG. 2—Calculated Change in Reflectivity after Soiling for Monochromatic Light

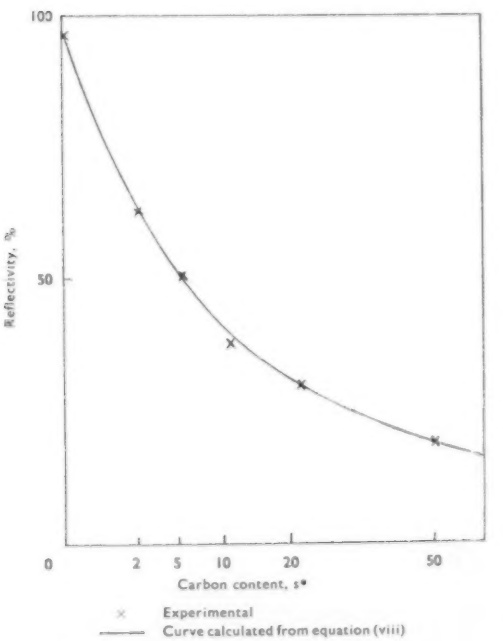

FIG. 3—Calculated and Observed Reflectivities for Undyed Starch

* Abscissae proportional to $s^{\frac{1}{2}}$

We conclude that agreement between predicted and experimentally found reflectivities of the soiled samples is very good.

4.6. CALCULATION OF Y VALUES

The calculation of the apparent brightness Y of a surface from the spectral reflection curve is a

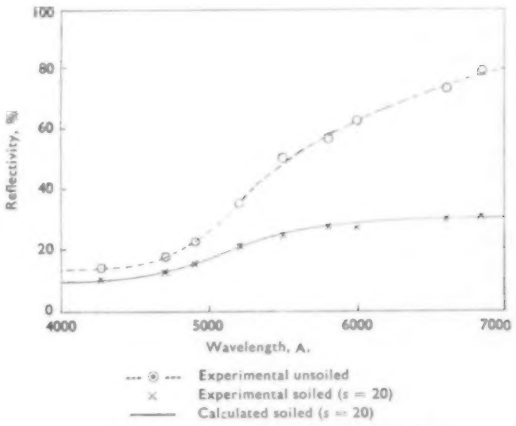

FIG. 4—Reflectance Curves for Lemon Sample

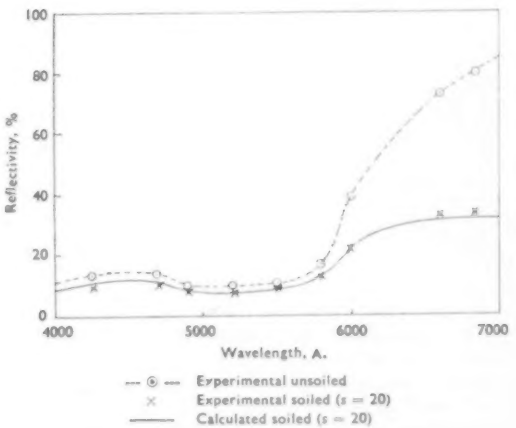

FIG. 5—Reflectance Curve for Claret Sample

standard procedure in applied optics. The calculation can be made for the apparent brightness of the surface in any illuminant for which the spectral distribution of energy can be specified. It was decided for present purposes to calculate Y_E, the brightness of the surface for an illuminant of equal energy at all visual wavelengths; this was done to derive a compromise figure for Y between that for a tungsten illuminant on the one hand and that for North daylight on the other. In practice Y is computed by summing the contribution $\bar{y}$ for each small region of the spectrum; it was found by trial and error that a sufficient accuracy in the calculation could be achieved by breaking up the visible spectrum into 16 regions each 200 A. wide. This procedure may introduce a little error into the computation of Y_0, but the corresponding error in Y_{20} will be such that H_{20} will be accurate to within 1%. The $\bar{y}$ values employed are given in Table XI, Appendix III. These were derived for the values of $\bar{y}_E$ tabulated in the *Handbook of Chemistry and Physics* [1].

A detailed computation of Y_0 and Y_{20} for the lemon sample (Colour 2) is set out in Table XII. Similar calculations were made on all the other

colours, and H_{20} was derived according to (ii). All these calculated brightness data are collected in the second part of Table III.

In Table IV the calculated order of soilability expressed as H_{20} is compared with the mean order found by a group of observers. Spearman's rank-correlation coefficient γ between the two orders is 0·98: i.e. the agreement is virtually complete. The essential difference is the reversal of the order of Samples 4 and 8. We believe that this is due to normal experimental error in preparing the soiled samples.

We have now reached a stage in the treatment of the data at which we have successfully achieved the first three objectives set out in § 2.3.

4.7. SOIL-RESISTANCE MERIT RATING (SMR)

The final point is to relate the calculated relative change in brightness to a relative apparent soilability.

It is possible to compute the concentration of soil on a white sample which will give the same apparent soiling H_{20} as that calculated for a given dyed sample. This quantity s_w can be determined by interpolation in Fig. 3, or more precisely by the use of equation (viii). The ratio of the concentration of soil on the coloured sample ($s = 20$) to that required to give an equal apparent soiling on the white sample ($s = s_w$) we propose to denote as the SMR value (colour or soil-resistance merit rating) of the colour. This is therefore defined as—

$$\text{SMR} = \frac{20}{s_w} \qquad \text{(ix)}$$

If it is assumed that, in wear, the rate of accretion of soil is constant (i.e. that s increases linearly with time), then the ratio of the time taken by the coloured sample to reach the standard soiling condition to the time taken by a corresponding white sample to attain the same apparent soiling H is the SMR value. The greater this quantity, therefore, the slower will be the onset of visible soiling in a given environment.

The concept of SMR values is a practical approach to the problem of giving a numerical value to the varying apparent soilabilities of different colours. It is based upon assessment of the relative soilability at a fixed content of physical soil rather than at a constant degree of apparent soiling for each colour. It would perhaps be more logical to compute figures for the latter case, but we believe that the increase in information obtained thereby, over that given by the relative SMR values, is too small to warrant the considerably greater labour of computation entailed.

Calculated values of s_w and SMR are given in Table V for the white starch and all the other eight colours. It is clear that the expected low soilability of dull and dark colours is reflected in the figures of the table, but we think that the magnitude of the effect is quite unexpected. Most medium depths have a soilability of only one-tenth or less of that of the white, and the darkest colour investigated, namely the blue (Colour 8), is one-seventieth as soilable as the white. The implications of this great range of relative soilings are very important in the design of carpets intended for heavy duty.

TABLE V

SMR Values of the Samples investigated

	Colour	s_w	SMR
1	White	20	1·0
2	Lemon	5·5	3·6
3	Crushed strawberry	1·2	16
4	Claret	2·2	9·1
5	Saxe blue	0·59	33
6	Almond green	1·5	14
7	Silver grey	3·3	6·1
8	Mineral blue	0·30	70
9	Terra cotta	1·5	14

5. Application of Optical Conclusions to Coloured Textiles

5.1. GENERALISATION OF THE RESULTS TO FIBROUS MATERIALS

The monochromatic reflectance of a textile can usually be adequately represented by the reflectance of an equivalent uniform coloured turbid solid. Nolan [2] and Pineo [3] have shown that, for dyed materials—

$$P = \frac{(1 - R)^2}{2R} = \frac{K}{S}$$

$$K = K_0 + ck_d \qquad \text{(x)}$$

where R is the fractional reflectance, S the coefficient of scattering of the colourless material, K the total coefficient of absorption, K_0 the coefficient of absorption of the undyed fibre, k_d the specific absorption of the dye, and c the concentration of the dye.

The present results on coloured starch may be shown to agree quite well with an extension of this principle—

$$P_{d,s} = \frac{K_0 + ck_d + sk_s}{S} \qquad \text{(xi)}$$

where k_s is the specific absorption of the dispersed carbon soil. It is interesting to note that soil particles, well dispersed but confined to the surface of the particles, simulate the behaviour of a uniformly distributed dye.

Equation (xi) may be rewritten—

$$P_{d,s} = P_d + \frac{sk_s}{S}$$

$$= P_d + \theta_s \qquad \text{(xii)}$$

where—

$$\theta_s = \frac{sk_s}{S} \qquad \text{(xiii)}$$

θ_s may be termed the *reflectivity decrement* of the soil present.

All that is known of the correspondence of the turbid-solid model with the behaviour of granular and fibrous material suggests that (xii) and (xiii) are immediately applicable to fibrous materials, if the appropriate scattering coefficient S of the uncoloured material is substituted.

5.2. DEPENDENCE OF SOILABILITY ON FIBRE TYPE

In relating apparent soiling to colour, the concept of SMR will be most useful with work clothes, permanent upholstery, and carpets. We propose here to consider only the implications in respect of carpets—

(*a*) On a given fibre in a given environment, apparent soiling will be the less the higher the SMR value of the colour of the surface fibres.

(*b*) With a given fibre of constant SMR, equation (xiii) indicates that apparent soiling is directly related to the soil content. Factors which decrease the rate of accretion of soil are obviously favourable, the most important being use of smooth-surfaced fibres, reduction of oily or greasy impurities to a low level ($< 0{\cdot}3\%$), and use of soil-retardants, of which the most successful are the "white soils" based on colloidal silica or alumina.

(*c*) With a fibre of given type, fineness, and colour, increasing dullness (produced, say, by spinning in a titania pigment) leads to a diminution in apparent soilability, since S in (xiii) increases. In our experience with carpets in actual wear this prediction is fully confirmed, and is in accord with the findings of Schappel [4] and Weatherburn and Bayley [5].

(*d*) As the filament size decreases, S increases proportionally to the surface area. Assuming that the accretion of soil particles is proportional to the relative surface area, then the apparent soiling rates of materials made from similar fibres of constant colour should be independent of the denier of the constituent fibres. This is in agreement with general experience, although precise experimental data are lacking.

5.3. FLOOR TRIALS

Floor trials with self-coloured carpets made from wool and viscose rayon, cellulose acetate, and regenerated protein staple fibres have shown that apparent soilability is closely related to SMR values of the pile fibres. Results may be classified—

Colour Behaviour	SMR Range
Unserviceable	1–3·9
Suitable for light duty	4·0–7·9
Moderately serviceable	8–15
Generally suitable	16–31
Suitable for heavy duty	32+

It may be noted that these results apply to carpets of uniform coloration. With colour-patterned carpets the overall impression of soiling is much reduced, although individual parts of the pattern may be seen on close inspection to be heavily soiled. Well broken areas of colour of fairly low SMR may be used in a patterned carpet without undue risk of complaint of rapid soiling.

It has already been said that the SMR values found for various colours must indicate greater differences in apparent soilability than is generally realised. In the floor trials the differences found between, say, a white or primrose yellow and a bright blue could only be described as startling, even with a prior knowledge of the respective SMR values.

The results quoted in the first paragraph above imply that the rates of soiling are similar, at a constant colour, for wool and the other manufactured staple fibres. This has been repeatedly confirmed, and average differences between the various fibres are much smaller than the differences due to colour.

5.4. SMR VALUES FOR WOOL DYEINGS

All typical carpet wools that we have seen undyed are a medium yellowish grey having only 50–60% of the reflectivity of a white viscose rayon pile. This original colour of the wool can be considered as optically equivalent to a grey dye on a viscose rayon fibre. On purely optical grounds, therefore, we would expect that any colour, even the brightest possible, dyed on to a wool carpet yarn should have a relatively low apparent soiling rate, equivalent to an SMR on viscose rayon of 4 or more. Thus it is clear that extremely sensitive colours are not dyed on wool because of the intrinsic nature of the wool fibre. We believe that this optical factor may contribute to the significantly better reputation of wool over viscose rayon in respect of soiling.

5.5. CHANGE OF HUE ON SOILING

Soiling with a neutral-coloured soil such as Carbon Black can cause a change in the dominant hue of the dyed material. It was early noticed in our experiments that the lemon (Colour 2) became distinctly greenish on soiling with Carbon Black pigment. It is generally agreed amongst colour experts that change in hue is more obvious than change in depth or saturation. We are of the opinion, however, that the flattening of the colour is so overwhelmingly greater than the small change in hue noted that it is flattening rather than hue change which is appreciated in the subjective assessment of soiling.

The change in hue noted is a necessary optical consequence of the relatively greater influence of the spectral regions of high reflectivity on the changes produced in the reflectance curve by soiling. Reference to Fig. 4 makes this plain for the lemon sample discussed above. Hue changes have been noted only with yellow, orange, and bright pink dyeings.

We conclude that change of hue on soiling, whilst interesting, is of no great significance generally in the consideration of apparent soiling rates of dyed textiles.

APPENDIX I

6. Details of Preparation of Dyed and Soiled Starch Samples

6.1. MATERIALS

(*a*) CORN STARCH—Brown & Polson's cornflour

(*b*) CARBON BLACK—Philblack O (Phillips Petroleum Co.).

6.2. DYED SAMPLES

The dyes used to obtain colours similar to those expected to be met in the carpet trade were in all cases direct dyes (Table I). To prevent undue swelling and consequent distortion of the starch granules, dyeing was carried out from a dyebath containing at least 70% ethanol by volume. All the dyes used were soluble in a dyebath of this composition. The strong dye solution was contained in a 2-litre three-necked round-bottom flask

equipped with a condenser, thermometer, and efficient mechanical stirrer. Dyeing was carried out on a boiling water-bath.

Having attained the desired colour by trial and error, the dyed starch was filtered off, washed with cold ethanol, and dried in a current of warm air.

In most cases the required colour was obtained fairly readily, although some difficulty was experienced in attaining the required depth with the heavier dyeings such as the crushed strawberry and almond green. Samples (100 g.) of each colour were prepared.

After dyeing and drying, it was found essential to grind the dyed starch lightly in a mortar and then to pass it through a fine nylon bolting cloth (10N quality) in order to break down the aggregates into which the starch granules had formed during dyeing and/or drying. In order to facilitate this sifting operation, which otherwise would be laborious, a small electrically driven rotary-drum type of sifter was constructed from Meccano. The drum was $3\frac{3}{4}$ in. in diameter, 4 in. wide, and thus sufficiently large to accommodate a charge of 100 g. with ease. It was found to be essential to add about 20% by volume of clean coarse silica sand to the charge in the drum in order to achieve a practicable rate of sifting. The inclusion of this coarse sand led to a very great increase in the rate of sifting, presumably because it caused movement in the layer of starch in contact with the gauze.

After this sieving operation, the dyed starch was in the form of a free-flowing fine powder and was ready for the subsequent preparation of the soiled samples.

6.3. SOILED SAMPLES

Having prepared the series of dyed corn-starch batches, the next operation was to soil these to given extents. Preliminary trials indicated that a suitable range of soiled samples would contain 2, 5, 10, 20, and 50 parts of Carbon Black per 10,000 parts of starch. This series was to be produced for each colour.

In preparing the range of soiled samples mentioned above, it is clear that it would be difficult to weigh out directly the very small quantities of pure carbon required: it was decided to prepare a series of stock mixtures of carbon and starch, from which the final mixtures could be obtained. For this purpose a mixture containing 10 g. of carbon per 100 g. of undyed starch was made up and used as the primary stock mixture, and from this, secondary stock mixtures were prepared, using the appropriate dyed starch in each case. These secondary mixtures all contained 1 g. of carbon per 100 g. starch. It will be appreciated that these mixtures necessarily contained both white and dyed starch, but the proportion of white starch in the final mixture was small and almost negligible. The proportion of white to coloured starch was, however, kept constant in all the final mixtures by adding the appropriate amount of excess white starch as required. The final mixtures were made up by dilution of the secondary stock mixtures.

6.4. MILLING

The initial mixing experiments demonstrated most dramatically that great care is required to disperse the carbon uniformly throughout the starch. The final somewhat laborious technique was adopted only after it had become apparent that simpler procedures were inadequate, since they produced samples in which the apparent soiling due to a given amount of carbon was much less than that found when dispersion was complete.

Simple dry mixing showed immediately that, no matter how long this mixing was continued, the resulting mix was inhomogeneous, large black specks being clearly visible in the pale grey background.

Recourse was then made to dry milling. For this operation $\frac{5}{8}$-in. steel balls were added to the mixture contained in a small screw-capped glass bottle, and the bottle was rotated on driven rollers. This milling gave rather better results than simple mixing, but it was not until the mix was made into a slurry with petroleum ether and then milled that a really satisfactory dispersion of the carbon was attained. Even with this technique the quality of the resulting mix was found to depend markedly upon the duration of the milling. Milling for 3 hr. was necessary to produce an essentially equilibrium condition, as judged by constancy of reflectance.

It was therefore decided that all millings should be carried out for 4 hr. under the following conditions—

Size of bottle	50 ml.
Number of $\frac{1}{4}$-in. steel balls	5
Weight of charge	10 g.
Volume of petroleum ether	12 ml.

It is emphasised that such milling is essential at every stage of dilution, and in the present work all mixings were treated in this manner. After milling, the petroleum ether was allowed to evaporate in a current of warm air. The resulting soiled starch was then obtained as a fine free-flowing powder.

APPENDIX II

7. Optical Method and Results

7.1. THE EEL PHOTOELECTRIC REFLECTOMETER

The EEL photometer with the reflectance spectrophotometer head is a cheap and robust instrument which appears to be very suited to the present series of experiments. We have found it reliable and capable of giving highly reproducible results. In repeat measurements we expect reproducibility to within 0·25% of full-scale deflection. We have found no indication that the instrument scale departs seriously from linearity, and have not found it necessary to apply the makers' small calibration corrections.

7.2. MEASUREMENT OF REFLECTANCE OF STARCH SAMPLES

A cell was made by cementing two $\frac{1}{2}$-in. sheets of Perspex (ICI) together, in the upper of which a 1-in. hole had been bored, thus forming a clean measuring cell 1 in. in diameter and $\frac{1}{2}$ in. deep.

It was found that, if the cell were filled with the starch sample, lightly tapped down, and the excess scraped away with a straight-edge, a very reproducible reflectance was obtained. All our measurements on starch have been made with this cell.

7.3. *Y* VALUES

The EEL photometer is provided with a set of tristimulus filters whereby the X, Y, and Z values can be obtained from three single readings on the sample. It is virtually impossible to procure a combination of filter and photoelectric cell which will give a completely satisfactory value, but checks made of the EEL instrument by comparison of measured X, Y, and Z values with those calculated from the spectral curves show that the instrument does indeed provide a good approximation to the correct values. Since we are concerned with the relative loss in brightness (i.e. the relative drop in Y value), the error for a particular colour will be similar to the error for the same colour after soiling; while the Y values themselves may be subject to a small uncertainty, the H value derived from them is probably more reliable, with a probable error of less than one unit.

The EEL instrument is designed to give the X, Y, and Z values of the surface for tungsten illumination.

7.4. PHOTOMETRIC RESULTS

A selection of detailed data are set out in Tables VI–IX.

TABLE VI

Table of Dominant Wavelengths of Spectral Filters of EEL Reflectometer

Filter	Wavelength (A.)
601	4260
602	4700
603	4900
604	5200
605	5500
606	5800
607	6000
608	6600
609	6840

TABLE VII

Reflectances (R %) for Unsoiled Starch Samples

No.	Sample Colour	Reflectance (%) for Filter 601	602	603	604	605	606	607	608	609
1	White	95·2	96·2	96·0	95·8	96·3	96·7	97·1	97·3	97·1
2	Lemon	14·3	17·9	22·2	35·7	51·0	57·6	63·7	74·2	79·0
3	Crushed strawberry	10·5	11·8	7·9	8·2	8·2	10·0	24·5	61·3	74·5
4	Claret	13·5	13·9	9·3	9·8	10·4	16·2	38·9	73·2	79·5
5	Saxe blue	24·9	24·9	18·0	15·0	11·0	8·5	10·0	17·8	26·5
6	Almond green	18·0	22·6	24·9	27·2	25·0	20·1	18·6	24·9	33·2
7	Silver grey	38·9	40·5	38·2	37·2	34·8	34·8	38·8	52·3	60·0
8	Mineral blue	12·2	12·2	10·2	10·0	8·9	7·9	8·1	9·2	10·0
9	Terra cotta	12·9	14·1	11·0	11·8	14·1	19·9	32·5	56·0	63·9

TABLE VIII

Reflectometer Y Values (%) for All Samples

No.	Colour	s ... 0	2	5	10	20	50
1	White	96·7	64·0	51·6	38·1	29·9	18·7
2	Lemon	51·5	44·8	38·5	32·5	24·3	13·9
3	Crushed strawberry	12·2	—	—	—	9·4	7·5
4	Claret	18·0	16·8	15·4	14·3	11·8	8·3
5	Saxe blue	11·0	10·7	10·7	10·4	9·1	7·2
6	Almond green	22·0	—	19·5	18·1	16·1	11·4
7	Silver grey	37·2	36·1	33·7	30·3	25·7	17·9
8	Mineral blue	8·4	8·0	7·8	7·3	7·4	6·0
9	Terra cotta	19·0	—	17·7	16·9	14·0	11·8

TABLE IX

Spectral Reflectance (R %) for Sample 2 (Lemon) and Sample 4 (Claret)

Filter Sample ...	601	602	603	604	605	606	607	608	609
2_0	14·3	17·9	22·2	35·7	51·0	57·6	63·7	74·2	79·0
2_2	14·0	17·0	21·4	33·3	45·7	50·8	54·3	60·2	62·9
2_5	12·9	15·9	20·8	30·8	40·6	44·0	46·3	50·7	52·9
2_{10}	11·7	14·3	18·2	26·4	33·4	36·0	37·9	41·0	42·9
2_{20}	10·4	12·8	15·9	21·2	23·5	26·9	27·9	30·1	31·6
2_{50}	7·9	9·1	10·6	13·1	14·9	15·3	16·1	17·1	18·1
4_0	13·5	13·9	9·3	9·8	10·4	16·2	38·9	73·2	79·5
4_{20}	8·3	9·4	7·6	7·9	8·9	12·7	21·9	32·3	34·5

7.5. SPECTRAL CURVES

The reflectance data, such as those given in Table VII, are plotted against wavelengths for each sample. A smooth curve is then drawn through the points, from 4000 to 7000 A. The 16 reflectances for the whole spectrum are interpolated at intervals of 200 A. for use in the optical calculations detailed in the next section.

TABLE X

Calculated Change in Monochromatic Reflectance for Soiling ($s = 20$)

R_0	R_{20}	R_0	R_{20}
5	4·5	55	27·0
6	5·3	56	27·3
7	6·1	57	27·5
8	6·9	58	27·7
9	7·6	59	27·9
10	8·3	60	28·1
11	9·0	61	28·3
12	9·6	62	28·5
13	10·3	63	28·7
14	10·9	64	28·9
15	11·5	65	29·0
16	12·1	66	29·2
17	12·7	67	29·4
18	13·3	68	29·5
19	13·8	69	29·6
20	14·4	70	29·7
21	14·9	71	29·8
22	15·4	72	29·9
23	15·9	73	30·0
24	16·3	74	30·1
25	16·7	75	30·2
26	17·1	76	30·3
27	17·6	77	30·4
28	18·0	78	30·5
29	18·5	79	30·5
30	18·9	80	30·6
31	19·3	81	30·6
32	19·7	82	30·7
33	20·1	83	30·6
34	20·4	84	30·6
35	20·8	85	30·6
36	21·2	86	30·5
37	21·5	87	30·4
38	21·9	88	30·4
39	22·2	89	30·3
40	22·6	90	30·3
41	23·0	91	30·2
42	23·3	92	30·2
43	23·7	93	30·1
44	24·0	94	30·1
45	24·3	95	30·0
46	24·6	96	29·9
47	24·9	97	29·8
48	25·2	98	29·7
49	25·4	99	29·5
50	25·7	100	29·3
51	26·0		
52	26·3		
53	26·5		
54	26·8		

7.6. FIBRE SAMPLES

Reproducible reflectance measurements on coloured textile materials are best obtained by cutting the sample into separate fibres about 1 mm. long. The cut fibres are suspended in water or suitable organic liquid as is appropriate, and the slurry compacted by filtration. If care is taken to achieve a pad thickness giving complete opacity, reproducible values of the reflectances can be determined on quite small samples. The one pad used with the various filters of the reflectometer affords directly all the data required for the preparation of the spectral reflectance curve.

APPENDIX III

8. Optical Computations

8.1. MONOCHROMATIC SOILED REFLECTANCES

The soiled reflectance is computed from the experimental reflectance by the following steps—

(*a*) Compute N_s from R_0 (iii)

(*b*) Compute N_s by (vii)

(*c*) Compute R_s from N_s (iii).

Table X gives R_{20} so computed for values of R_0 from 5 to 100 by steps of 1. In practice, therefore, the required value of R_{20} may be taken from the table for each of the 16 values of R_0 interpolated from the experimental spectral reflectance curve.

8.2. CALCULATION OF Y_0 AND Y_{20}

The visible spectrum is divided into sixteen regions each 200 A. wide; the mean brightness coefficient for an equal-energy source $\bar{y}_E$ for the centre of each region is given in Table XI.

TABLE XI

Apparent Brightness of Equal-energy Spectrum Regions for a Standard Observer

Wavelength (A.)	$\bar{y}_E$
4000	0·001
4200	0·005
4400	0·023
4600	0·061
4800	0·140
5000	0·330
5200	0·704
5400	0·950
5600	0·991
5800	0·867
6000	0·631
6200	0·381
6400	0·177
6600	0·062
6800	0·017
7000	0·006

$$\Sigma \bar{y}_E = 5{\cdot}346$$

A typical calculation of Y_0 and Y_{20} is given in Table XII. It proceeds by the steps—

(*a*) In column 2 set out the interpolated reflectances R_0 for each of the wavelengths of column 1. The interpolation is made as described in § 7.5.

(*b*) In column 4 set out the corresponding reflectances R_{20} taken from Table X.

(*c*) In columns 3 and 5 set out the products of R_0 and R_{20} respectively with the corresponding $\bar{y}_E$ of Table XI.

(*d*) Sum columns 3 and 5 to give respective $\Sigma\bar{y}$; divide these sums by $\Sigma\bar{y}_E$ (5·346) to give Y_0 and Y_{20} respectively.

8.3. CALCULATION OF THE SMR VALUES

(*a*) Compute H_{20} from Y_0 and Y_{20} (ii)

(*b*) By means of equation (viii) for undyed starch compute s_w, the concentration of soil which will give the same H_s as found in (*a*) above.

(*c*) Compute SMR by (ix).

TABLE XII

Sample Calculation of Y_{20} and H_{20} for Colour 2 (Lemon)

Wavelength (A.)	R_0 %	$\bar{y}_0$	R_{20} %	$\bar{y}_{20}$
4000	14·0	0·000	10·9	0·000
4200	14·2	0·001	11·0	0·001
4400	15·2	0·004	11·6	0·003
4600	16·5	0·010	12·4	0·008
4800	19·5	0·027	14·1	0·020
5000	25·5	0·084	16·9	0·056
5200	35·7	0·251	21·1	0·149
5400	46·5	0·442	24·8	0·236
5600	53·8	0·533	26·7	0·264
5800	57·6	0·499	27·6	0·239
6000	63·7	0·402	28·8	0·182
6200	68·0	0·259	29·5	0·112
6400	71·2	0·126	29·8	0·053
6600	74·2	0·046	30·1	0·019
6800	77·7	0·013	30·5	0·005
7000	83·0	0·005	30·6	0·002

$\Sigma\bar{y} = 2{\cdot}702$ $\quad \Sigma\bar{y} = 1{\cdot}349$

$Y_0 = 50{\cdot}5\%$ $\quad Y_{20} = 25{\cdot}2\%$

$H_{20} = 50{\cdot}1$

SMR $= 3{\cdot}6$

TABLE XIII

s_w and SMR as a Function of H_{20}

H_{20}	s_w	SMR
5	0·027	740
6	0·040	500
7	0·053	380
8	0·071	280
9	0·090	220
10	0·11	180
11	0·13	150
12	0·16	125
13	0·19	105
14	0·23	85
15	0·27	75
16	0·32	60
17	0·36	55
18	0·41	49
19	0·46	43
20	0·52	38
21	0·58	34
22	0·65	31
23	0·72	28
24	0·80	25

TABLE XIII—*continued*

H_{20}	s_w	SMR
25	0·87	23
26	0·95	21
27	1·04	19
28	1·13	18
29	1·22	16
30	1·33	15
31	1·45	14
32	1·57	13
33	1·70	12
34	1·83	11
35	1·97	10
36	2·11	9·5
37	2·25	8·9
38	2·41	8·3
39	2·58	7·7
40	2·77	7·2
41	2·97	6·7
42	3·19	6·3
43	3·42	5·8
44	3·67	5·5
45	3·94	5·1
46	4·2	4·8
47	4·5	4·4
48	4·8	4·2
49	5·1	3·9
50	5·5	3·6
51	5·8	3·4
52	6·2	3·2
53	6·6	3·0
54	7·0	2·9
55	7·5	2·7
56	8·0	2·5
57	8·5	2·4
58	9·1	2·2
59	9·7	2·1
60	10·4	1·9
61	11·1	1·8
62	11·8	1·7
63	12·6	1·6
64	13·5	1·5
65	14·3	1·4
66	15·6	1·3
67	16·8	1·2
68	18·1	1·1
69	19·3	1·0

The results of such calculations are given in Table XIII, where values of s_w and SMR are recorded for H_{20} ranging from 5 to 69% by steps of 1.

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Optical Studies
II—SMR Values for a Comprehensive Range of Colours

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The SMR values of a wide range of colours have been measured and the results listed. Two series of tests have been made. The dependence of SMR on depth of colour and flattening is examined in detail. Reference to the listed values will enable any colour to be assessed approximately in respect of the relative liability to show soiling.

1. INTRODUCTION

The soil-resistance merit rating (SMR value) of a coloured textile material has been defined in Part I[1]; it characterises the relative apparent soilability of the colour by suitable computation from the spectral reflectance curve of the unsoiled material.

The SMR value of any coloured textile can be determined experimentally by the procedure described in detail in Part I. In many instances, however, it is convenient and sufficiently accurate to assess the SMR value approximately by comparison with readily accessible standard colours. We have therefore measured the SMR values of 78 standard colours taken from the Duracol† ranges on filament and staple viscose rayon, and have in in each case indicated the nearest B.C.C. equivalent colour. Reference, therefore, to the appropriate colour card or to the British Colour Council *Dictionary*[2] will provide adequate material for approximate assessment of SMR by matching.

We have also determined SMR values for six representative hues, each presented in six depths of dyeing crossed by six levels of greying, using coloured material given in the Scandinavian Colour Book[3]. Variation in SMR is here displayed in terms of the colourist's variables—depth of colour and flattening.

2. EXAMINATION OF A COMPREHENSIVE RANGE OF COLOURS

The reflectance curves of the Duracol samples were measured at a series of wavelengths between 4000 and 7000 A. using the EEL reflectance spectrophotometer head (Evans Electroselenium Ltd., Harlow). The samples of fibre were used in the form of pads of fibre chopped to approx. 1–2 mm. in length; care was taken to ensure that all the pads were effectively of infinite thickness in the sense that transmitted light is of negligible intensity; the SMR value was then calculated from the reflectance curve in the manner previously described (Part I).

The SMR values obtained in this way can be related to the expected relative soilability of the colour in wear according to the following classification—

Very poor	1–3·9
Poor	4·0–7·9
Fair	8–15
Good	16–31
Very good	> 32

TABLE I

Tabulation of SMR Values of Colours arranged in Dyers' Order

Duracol No. and Designation		B.C.C. No. and Name		SMR Value
		YELLOWS		
FD 19	Sulphur	BCC 112	Sulphur	1·9
SD 50	Yellow	BCC 5	Maize	2·1
SD 26	Gold	BCC 54	Saffron	2·9
SD 34	Gold	BCC 54	Saffron	4·0
FD 20	Indian yellow	BCC 6	Indian yellow	3·8
SD 14	Champagne	BCC 5	Maize	2·2
		ORANGES		
SD 67	Vanilla	BCC 142	Corn husk	2·4
SD 73	Apricot	BCC 143	Apricot	2·9
SD 2	Light peach	BCC 91	Salmon	2·6
SD 69	Tango	*BCC 58	Rust	9·7
SD 20	Rust	*BCC 58	Rust	14
SD 24	Peach	BCC 91	Salmon	3·2
		REDS AND PINKS		
FD 57	Peach	BCC 206	Shell pink	2·6
SD 72	Begonia	*BCC 125	Brick red	10
SD 68	Geranium	*BCC 125	Brick red	7·8
SD 83	Scarlet	BCC 210	Union Jack red	4·9
SD 59	Strawberry	BCC 182	Strawberry pink	4·3
FD 14	Red	BCC 185	Cherry	5·9
SD 1	P.O. red	BCC 186	Cardinal	8·7
SD 74	Regency rose	*BCC 157	Old rose	12
SD 60	Blossom pink	*BCC 183	Begonia	9·0
SD 28	Rose	BCC 183	Begonia	5·6

† *Duracol* is the registered trade mark used by Courtaulds Ltd. and British Celanese Ltd. to denote their spun-dyed yarns and fibres.

TABLE I—*continued*

Duracol No. and Designation		B.C.C. No. and Name		SMR Value
		REDS AND PINKS—*continued*		
SD 35	Pink	*BCC 182	Strawberry pink	5·7
FD 21	Pink	*BCC 34	Blossom pink	6·2
SD 70	Rose	BCC 158	Crushed strawberry	12
SD 57	Pink	BCC 14	Baby pink	3·0
SD 41	Rose	*BCC 157	Old rose	6·9
SD 66	Old rose	BCC 157	Old rose	9·8
SD 58	Amaranth pink	BCC 34	Blossom pink	11
SD 84	Ruby	BCC 37	Peony red	19
SD 80	Claret	BCC 159	Raspberry	24
SD 48	Red	BCC 159	Raspberry	29
		VIOLETS		
SD 38	Wine	BCC 39	Maroon	58
SD 25	Maroon	BCC 39	Maroon	65
FD 54	Maroon	BCC 160	Garnet	60
FD 101	Magenta	*BCC 107	Amaranth pink	21
		BLUES		
SD 56	Wedgwood	BCC 215	Wedgwood	7·7
SD 55	Cornflower	BCC 218	Union Jack blue	140
FD 16	Dark blue	BCC 219	Purple navy	140
SD 54	Victrix blue	BCC 47	Victrix blue	160
SD 49	Royal	*BCC 197	Royal blue	56
SD 19	Blue	BCC 196	Larkspur	20
SD 33	Blue	BCC 195	Delphinium	18
SD 22	Medium blue	*BCC 195	Delphinium	9·5
SD 79	Sapphire	BCC 88	Sapphire	120
FD 15	Medium blue	BCC 47	Victrix blue	130
FD 12	Light blue	BCC 45	Saxe blue	36
SD 89	Beau blue	BCC 84	Forget-me-not	5·1
SD 12	Light blue	*BCC 162	Sky blue	3·5
		BLUE–GREENS		
SD 64	Turquoise blue	*BCC 118	Turquoise	5·6
SD 39	Marina	*BCC 191	Cambridge blue	15
FD 56	Turquoise	*BCC 121	Turquoise green	7·4
		GREENS		
SD 53	Turquoise green	BCC 121	Turquoise green	3·5
FD 59	Hunter green	BCC 26	Tartan green	63
SD 78	Bottle green	BCC 25	Bottle green	59
SD 32	Green	BCC 10	Almond green	18
SD 30	Green	BCC 23	Malachite green	16
FD 18	Malachite green	BCC 24	Green beetle	37
FD 17	Apple green	BCC 22	Apple	14
SD 62	Emerald	*BCC 213	Emerald	17
SD 23	Medium green	BCC 99	Water green	4·0
SD 8	Nil green	*BCC 8	Lichen green	4·4
SD 77	Mist green	*BCC 9	Mistletoe	9·6
SD 42	Pale green	*BCC 21	Eau de Nil	5·1
SD 76	Olive green	*BCC 78	Olive green	21
		BROWNS AND BEIGES		
SD 40	Satinwood	BCC 65	Satinwood	5·4
SD 29	Old gold	BCC 66	Buff	11
SD 90	Walnut	BCC 68	Oakwood	18
SD 37	Copper	BCC 134	Horse chestnut	33
FD 52	Tan	*BCC 63	Ecru	4·7
FD 102	Warm brown	BCC 18	Rose beige	22
SD 86	Fawn	*BCC 168	Nutmeg	13
SD 71	Mushroom	BCC 18	Rose beige	21
FD 55	Brown	BCC 19	Coffee	190
SD 52	Dark brown	BCC 130	Falcon	97
		GREYS		
SD 47	Gunmetal	BCC 42	Gunmetal	25
FD 58	Slate	BCC 155	Graphite	28
		BLACK		
SD 11	Black	BCC 220	Jet black	ca. 450

* Dictionary colour not a good match.

The complete results obtained are given in Table I. There the 78 colours are listed in dyers' order of colour, together with the nearest equivalent in the British Colour Council *Dictionary of Colour Standards* (*B.S.* 543:1934). Poor matches have been marked with an asterisk.

TABLE II

Dependence of SMR on Depth of Colour and Flattening

Grey Value	Depth of Colour 20	30	40	50	60	70
	A—YELLOW 02					
20	1·9	1·9	2·0	2·1	2·3	2·5
30	4·3	4·4	4·5	4·6	4·7	4·9
40	9·4	9·5	9·6	9·7	9·9	10
50	22	22	22	22	22	23
60	46	47	47	48	48	49
70	>†	>	>	>	>	>
	B—ORANGE 12					
20	2·6	2·7	2·9	3·1	3·5	4·3
30	5·0	5·3	5·8	6·2	6·8	7·2
40	9·4	9·5	9·6	9·8	9·9	10
50	20	21	22	22	22	23
60	48	50	52	55	58	65
70	>	>	>	>	>	>
	C—RED 30					
20	3·0	4·7	5·3	5·8	6·8	8·1
30	6·1	7·5	9·9	10	11	13
40	12	16	17	19	20	22
50	23	27	35	40	45	50
60	55	80	>	>	>	>
70	>	>	>	>	>	>
	D—VIOLET 44					
20	2·8	4·3	6·5	10	16	25
30	5·2	7·0	10	15	23	32
40	10	13	17	23	31	41
50	22	27	33	42	54	69
60	46	51	59	68	80	100
70	>	>	>	>	>	>
	E—GREEN 82					
20	3·0	5·0	7·0	9·6	13	23
30	5·5	7·2	9·1	13	17	30
40	12	15	17	21	27	38
50	23	27	34	40	51	65
60	50	55	65	79	90	>
70	>	>	>	>	>	>
	F—BLUE 62					
20	3·5	17	35	81	>	>
30	7·0	22	40	83	>	>
40	16	33	55	97	>	>
50	30	45	100	>	>	>
60	65	>	>	>	>	>
70	>	>	>	>	>	>

G—GREY 00

Grey Value	
20	1·9
30	4·2
40	9·5
50	21
60	46
70	>

† Values greater than 100 have been indicated by >.

3. EFFECT OF DEPTH OF COLOUR AND FLATTENING

In order to study more fully the effect of these two important practical variables, use was made of the Scandinavian Colour Book[3]. In this colour card the colour gamut has been subdivided into 48 hues, each hue being represented by a separate card in the dictionary. Each hue is presented as 36 shades (in 6 depths and each in 6 degrees of greyness). Pictorially the system may be considered as a cylinder, each card representing a semi-axial section through the cylinder. The circumference of the cylinder represents the gamut, and the axis of the cylinder grey, with white at the top and black at the bottom; the radius of the cylinder accommodates increasing depth of colour.

Six representative colour cards were selected from the dictionary—yellow, orange, red, violet, blue, and green. The reflectance curves of all of the samples on each card were measured with the EEL reflectometer. From these reflectance curves the appropriate SMR values have been computed as before.

The results are given in Tables II A–F (with additionally II G, results for the grey scale) and illustrate the effect of depth of colour and flattening on SMR value. Apart from white, the colour most sensitive to apparent soiling is yellow; even with quite heavy dyeings the SMR is but little increased (SMR values < 3) unless the colour is flattened. Increasing greying of the colour increases the SMR value to an extent approximately equal to the effect of the grey component alone. The same conclusion is nearly true of bright orange, but with bright reds increasing depth of colour makes a significant contribution to increase in SMR value. Blue is the colour least sensitive to apparent soiling. Here the SMR increases with depth of colour even more rapidly than with increased grey content, so that quite high SMR values can be attained with moderate depth of colour and moderate flattening. Green and violet are intermediate in character between red and blue.

4. DISCUSSION

The following general conclusions may be drawn from present results—

(i) Medium or deeper shades of green, greenish blue, blue, violet, brown, beige, and grey should be satisfactory in showing a low relative apparent soiling rate.

(ii) Bright yellow, bright orange, or bright red, at all depths of colour, must be considered as highly soilable.

(iii) All yellows, oranges, and reds require considerable flattening to give a low apparent soiling rate.

(iv) White and all pastel colours will show a high relative apparent soiling rate.

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TEXTILE RESEARCH LABORATORY
BOCKING
BRAINTREE
ESSEX

(*Received 14th July 1959*)

References

[1] Mann, H. B., and Morton, T. H., J.S.D.C., **75**, 522 (Nov. 1959).
[2] British Colour Council, *Dictionary of Colour Standards*, *B.S.* 543:1934 (London 1934).
[3] *Scandinavian Colour Book*, 3 vol. (Copenhagen 1956).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

The Physical State of Direct Dyes in Viscose Rayon and its Influence on Light Fastness

Giles and co-workers[1–3] have postulated that water-soluble dyes, such as the direct dyes, exist at least in part as aggregates or crystals of submicroscopic size within dyed fibres. They further postulated that the variation in light fastness among water-soluble dyes on a given substrate arises more from differences in their physical (aggregation) state in the substrate than from differences in their chemical nature. The first objective in our study of the factors influencing the light fastness of dyes on cellulosic materials was to determine whether aggregation of direct dyes could be detected in cellulose with the electron microscope. To achieve this it was first necessary to prepare specimens of direct-dyed cellulose that would lend themselves to electron-microscopical examination. Since methods of ultrathin sectioning of dyed fibres had not yet been developed sufficiently for use in high-resolution electron microscopy, dyed ultrathin viscose films of about 500–1000 A. thickness were prepared and used as primary specimens for the electron microscope. The ultrathin films were prepared from viscose dope that was taken from the commercial batch about to be spun into rayon. A special technique and apparatus made it possible to dye the viscose films under conditions corresponding to those frequently used in commercial practice.

The cellulose, of which the viscose films are composed, is structurally similar to that of rayon fibres in almost every respect. In fact, the films differ from rayon only in that their polycrystalline polymer structure is unordered, whereas that of rayon fibres is partially ordered as a result of spinning and drawing. It could be reasonably assumed from this similarity that aggregation of the dye as observed in the viscose films would also occur in rayon dyed under the same conditions.

When this method was used to examine two direct dyes in viscose, their physical state was found to differ markedly. Aggregates of one dye could not be detected in the viscose films at a resolution limit of about 30 A., whereas aggregates of the other dye were plainly visible in the viscose films dyed under the same conditions. The purified dyes were applied to spun rayon fabric under conditions corresponding to those used to dye the ultrathin viscose films. When exposed simultaneously in the Fade-Ometer, the apparently unaggregated dye exhibited considerably poorer light fastness on rayon than the one which aggregated in viscose. This difference in light fastness could have resulted from either a difference in the physical state of the dyes in the fibres or the chemical nature of the dyes. Thus, in order to obtain further indication as to whether the physical state of direct dyes is important, this study was extended to a number of other direct dyes of diverse chemical structure, which were selected in an attempt to randomise this factor, while comparing the light fastness of the dyes on rayon with their physical state in viscose.

Only two of the nine dyes examined did not appear to aggregate in viscose. These apparently unaggregated dyes exhibited considerably poorer light fastness on rayon than the others, which aggregated sufficiently in viscose to be detectable with the electron microscope.

The results of this study indicate that many direct dyes exist at least partly in an aggregated state in viscose. They also support the idea that the physical state of direct dyes in viscose is important in determining their light fastness on this material. Full details of this work will be published in the *Textile Research Journal*.

L. Weissbein
Bound Brook Research Laboratories
Organic Chemical Division
American Cyanamid Co.
Bound Brook N.J.
U.S.A.

7th October 1959

[1] Baxter, G., Giles, C. H., McKee, M. N., and Macaulay, N., J.S.D.C., **71**, 218 (1955).
[2] Baxter, G., Giles, C. H., and Lewington, W. J., *ibid.*, **73**, 386 (1957).
[3] Giles, C. H., *ibid.*, **73**, 127 (1957).

Notes

Meetings of Council and Committees

October

Council— 7th

Finance and General Purposes— 12th

Publications— 20th and 27th

Fastness Tests Co-ordinating— 13th

Terms and Definitions— 2nd

Death

We regret to report the death of Mr. Wilfred Kershaw.

Chemical Society Library

The above library, which is available for use by members of the Society, will be closed from 1 p.m. Wednesday 23rd December to 9-30 a.m. Tuesday 29th December 1959.

B.S. 1006 : 1955

Readers may be interested to know that the Society's method for determining light fastness, which is published as *B.S.* 1006 : 1955, is being widely used by the British Standards Institution in the establishment of standards of performance, not only for coloured textiles, for which it was originally intended, but also for other substrates.

The list shows those British Standards of performance which employ the Society's blue wool standards.

Material	British Standard	Minimum Light Fastness
TEXTILES		
Linen textiles for use by hospitals and local authorities	1781 : 1957	5
Berets of knitted wool	1908 : 1952	5
Rayon dress and blouse fabrics	2022 : 1953	3
Rayon lingerie fabrics	2023 : 1953	2
Cotton velveteens for loose cushions	2802 : 1956	4
Woven upholstery fabrics	2543 : 1954	5
Cotton tickings for hospitals	2732 : 1956	5
Cotton furnishing fabrics	3153 : 1959	4–7 according to type
NON-TEXTILES		
Anodised aluminium	1615 : 1958	3 (interior) 7 (exterior)
Thin PVC sheeting	1763 : 1956	5
Leathercloth for upholstered furniture	2601 : 1955	5
Letterpress four-colour printing inks	3020 : 1959	4
Filters for protection against intense sun glare	2724 : 1956	5
PVC insulation and sheaths for electric cables	2746 : 1956	4
Cables for vehicles	1862 : 1952	4
PVC insulated cables and flexible cords	2004 : 1955	4
Polystyrene tiles for walls and ceilings	2552 : 1955	> 4
Flexible PVC extrusion compounds	2571 : 1955	4
Thick PVC sheeting (flexible, unsupported)	2739 : 1956	5
Cellulose acetate moulding material	1524 : 1955	Subject to agreement

New Books and Publications

The Emergence of the German Dye Industry

By John Joseph Beer. (*Illinois Studies in the Social Sciences*—Volume **44**.) Pp. vii + 168. Illinois: University of Illinois Press. 1959. Price, paper $3.50, cloth $4.50.

This very readable account of the establishment of the German dye industry is based essentially on secondary sources, apart from the unpublished papers of C. Duisberg, B. Heymann, and O. Doermer. These sources are covered fairly completely except that Mr. Beer ignores the re-examination of the beginnings of the British industry which was stimulated by the Perkin Centenary as he does Haber's subsequent *Chemical Industry during the Nineteenth Century* and the work of Padley, Hardie, and Clow on the origins of the chemical industry, all of which is relevant to his thesis. Cliffe's study of Griess was published when this work was previously in the press but there are earlier papers of Cliffe and Ward on Griess, the retirement of Perkin from the industry, on Mansfield and Crace-Calvert which a scholarly survey can scarcely overlook.

Four out of Mr. Beer's first five chapters cover the beginning of the dye industry in Great Britain and its subsequent decline, the remaining one of these chapters dealing with the decline of the French colour industry—incidentally doing less than justice to Roussin and Lauth. The next seven chapters describe the beginning of dye manufacture in Germany and Switzerland, the influence of the university and the polytechnical institute—a chapter which could well be read together with a corresponding one in Dr. Lilge's study of the German universities in *The Abuse of Learning*—the rise of the industrial research laboratory, the construction of the German sales organisation and empire, the relations between the dye industry and the German state, the formation of the cartel and the dye industry during and after the first world war. From these chapter headings it will be seen that Mr. Beer has grasped the significance of the essential factors in the rise of the German industry.

Nevertheless, this is a descriptive rather than a critical account of the rise of the German industry. The reference to Alizarin in the chapter on cartel formation is inadequate and should be supplemented by some account of the circumstances which impelled the Turkey Red dyers of this country to form the British Alizarine Company. He notes Ivan Levinstein's familiarity with German methods in the comparative success of that British firm but Levinstein's practice of importing peripatetic German chemists could also have played

some part. The first two Ivan Levinstein Memorial Lectures could well have found place in his references. Again, the passing reference to the formation of the British Dyestuffs Corporation is inaccurate and the two sentences containing it require both correction and expansion. There are a few bibliographical errors and the indexing is open to criticism at points, but these are minor blemishes in a balanced narrative which reiterates from a different point of view lessons which were explained three years ago at the Perkin Centenary but which have still to be fully grasped in British industry.

R. BRIGHTMAN

Steric Effects in Conjugated Systems

Proceedings of a Symposium held at The University, Hull, 15–17th July 1958, by the Chemical Society

Edited by G. W. Gray. Pp. viii + 181. London: Butterworths Scientific Publications. 1958. Price 30s. 0d.

Of the fifteen papers, seven are concerned with ultraviolet absorption spectra, and of these, two attempt to interpret the observations and predict the shifts of the absorption maxima in molecular orbital terms. Four papers report the results of kinetic measurements on solvolysis, substitution, hydrolysis and esterification, and hydrogen–deuterium exchange. Single papers on the effects of substitution on dipole moments, anisotropic liquid phases, and vapour-phase chromatographic adsorption, together with a brief historical introduction, complete the record.

Although a good deal is already known about steric hindrance, these papers, and particularly the discussions, emphasise how much remains to be discovered before we can obtain a full understanding of the subject. For example, it is known that the introduction of substituents which force conjugated systems out of planarity will cause the relevant absorption bands to diminish in intensity, but the wavelength shift, though generally hypsochromic, still cannot be forecast with certainty.

The book is well produced and is gratifyingly free from misprints. Being a record of papers presented by specialists to specialists, this volume will have rather a limited appeal, but it should provide the worker connected with dye-research with food for thought. There are no indexes, but this will not deter the specialists at whom it is aimed.

E. V. TRUTER

Heterocyclic Chemistry

An Introduction

By Adrien Albert. London: University of London, The Athlone Press, 1959. Pp. x + 424. Price, 45s. 0d.

The field of heterocyclic chemistry has been for the last decade or so in a state of almost explosive expansion, noticeable even against a background of general expansion in all fields of organic chemistry. The main impetus certainly springs from the fact that so many biologically active substances, both natural and synthetic, are heterocyclic. A growing stream of papers in this field emerges especially from the laboratories of the drug houses, of medical research units, and, of course, of the universities. Existing single-volume works on heterocyclics are inadequate and out of date. The present volume, therefore, fills an obvious gap, and it is appropriate that the author should hold a chair of medical chemistry (at the Australian National University, Canberra).

To cover so diverse and ramified a topic as heterocyclic chemistry in one slim volume in the classical textbook sense is impossible: Professor Albert has adopted an interesting and effective scheme for containing his material. Factual dogmatic presentation is reduced to a minimum, and effort is concentrated on correlating reactivity with electronic structure in such a way that general regularities and trends are shown. He has divided the heteroaromatics into those with a deficit of π-electrons on the carbon atoms (π-deficient, e.g. pyridine) and those with an excess (π-excessive, e.g. pyrrole). His main treatment is contained in chapters on Heteroparaffinics, General Discussion on Heteroaromatics, π-Deficient *N*-Heteroaromatics, π-Excessive *N*-Heteroaromatics, π-Excessive *O*- and *S*-Heteroaromatics, and Heteroethylenics (i.e. non-aromatic unsaturated heterocyclics). In each chapter on heteroaromatics a first section briefly introduces the parent substances and outlines nomenclature, a second section deals with the correlation of structure and properties, and a third section presents what the author terms "monographs", i.e. brief sketches of individual compounds or groups, with details of syntheses and applications. In the correlative sections the author concerns himself with the interpretation of the physical properties and reactions of his different groups in terms of electronic statics and dynamics. "Net-charge" diagrams serve as a basis for much of the discussion. The chapters on Heteroparaffinics and Heteroethylenics follow more conventional lines. The rest of the book comprises chapters on Spectra, Ionisation Constants, Oxidation–Reduction Potentials and Dipole Moments, Interpretation of Complex Formulae in terms of Physical and Chemical Properties, and Notes on some Rational Approaches to new Syntheses. Throughout the author does not take the fundamental physical principles for granted: he concisely sketches in the relevant theory in every case.

The author is content to leave detailed accounts of particular heterocyclic types to others; for each type he quotes appropriate reference works or review articles. Naturally enough, such digressions from the main theme as he permits himself are mainly in the biochemical and pharmacological fields. He attempts to deal honestly with uses of heterocyclics as dyes and pigments, but limitations of space permit only the most skeletal and general treatment. For example, pyrazolone dyes are despatched in 11 lines, cyanine dyes in 9 lines, and phthalocyanine pigments in 6 lines. Natural colouring matters are given scarcely more room: the anthocyanidins and the haem pigments each receive about 1½ pages. Some minor criticisms are mainly the result of such compression. The author is very frugal with reaction schemes. Careful

reading involves much referring forward and backward, and the reviewer felt more than the average impatience with the inevitable Roman formulae numbers (one chapter reaches CXXXVII).

This work is not a textbook; it is something richer and rarer. It is doubtful whether the average undergraduate would welcome it. The lack of dogmatism, the wealth of correlation and argument, and the absence of preparations and properties, duly numbered, make it a swotter's nightmare. Nevertheless, if he could be persuaded to read appropriate sections he would gain an understanding of heterocyclics which the average textbook would not give him. Professor Albert's book is a research worker's manual and source-book. So judged it deserves the highest praise. It is a most stimulating and idea-provoking work, and will find a place of honour in the libraries of most workers who deal with heterocyclic compounds. The chapters on physical properties (VIII–XI) are particularly valuable despite their brevity. Lastly, but most valuable of all and illustrative of the size of the author's task, no fewer than 1,035 references to original papers, reviews, and monographs are quoted.

D. G. LEWIS

Hydrogen Bonding

Papers presented at the Symposium on Hydrogen Bonding held at Ljubljana, 29th July–3rd August 1957

Edited by D. Hadži and H. W. Thompson. Pp. xii + 571. London: Symposium Publications Division, Pergamon Press Ltd. 1959. Price 110s. 0d.

Frankland, then Professor of Chemistry at Owens College, Manchester, in 1852 first formulated the idea of the valency of atoms. Hydrogen was then recognised as behaving always as a typical univalent element. For sixty years apparently no facts were discovered to throw any doubt on this belief. Then in 1912 Moore and Winmill, at Oxford, made a tentative suggestion that the properties of trimethylammonium hydroxide might be best explained by assuming that one hydrogen atom could be joined to two other atoms at the same time, and in the following year Pfeiffer in Zürich made a similar suggestion to account for some properties of lake-forming coloured substances. In the years immediately following, the electronic theory of valency was first developed, by Langmuir, Lewis, and Kossel, and this enabled Latimer and Rodebusch, and also Huggins, in 1920, working in California, to explain how a second valency bond can be formed by hydrogen, and to offer the first suggestion of the modern conception of water as a system of many H_2O molecules interlinked by a second valency or "hydrogen bond".

The hydrogen bond was a revolutionary idea, accepted only slowly by chemists, until its existence was more clearly revealed by modern physical techniques, especially X-ray crystallography and infrared spectroscopy. The concept of this bond is now, however, recognised as one of the great fundamental principles in chemistry, capable of explaining and co-ordinating a vast amount of hitherto unexplained knowledge of the properties of chemical substances. Indeed, the properties of the essential constituents of all living matter—water, cellulose, proteins, nucleic acids—are now understood to be determined by the hydrogen bond acting in and between their molecules. This bond is responsible for many of the properties of fibres and their reactions with dyes, and some knowledge of its occurrence is clearly essential to all chemists in the textile and dyeing industries.

The present volume is a detailed record of nearly 60 papers delivered at an international conference. The emphasis here is largely on the physics of the bond and on the results of using modern physical methods for its examination, though a few papers deal with some aspects of the effects of the bond on the chemical properties of organic compounds, including their adsorption by fibres.

Most of the papers are in English. The production is of the first-class quality we associate with this publisher and with the printing house of Chorley & Pickersgill Ltd. The work will no doubt be accepted as a standard work on the subject for many years.

C. H. GILES

Handbook of Textile Fibres

By J. Gordon Cook. Watford: Merrow Publishing Co. Ltd. 1959. Pp. xxv + 422. Price, 15s. 0d.

This small book bravely attempts to deal with the properties of all the known fibres, natural and synthetic. The work will be extremely valuable to the technologist in the field.

The subject-matter is divided into sections on fundamental fibre structure, natural fibres of vegetable origin, natural fibres of animal origin, natural fibres of mineral origin, regenerated fibres, and synthetic fibres. The author has collected information not only on the common materials but also on the lesser known naturally occurring substances used as fibres; in this connection, the list of fibres produced from leaves is particularly interesting.

The short accounts of the history of the natural fibres such as wool and cotton make interesting reading, but in such a small book the space devoted to this could have been more valuably used. Thus the account of the fundamental structure is very terse, and would be helped considerably by the inclusion of a few diagrams.

The accounts of the properties of the separate synthetic and regenerated fibres are very well conceived, and the author has not wasted space with long accounts of possible end-uses. The lists of trade names and manufacturers of each type of fibre are valuable for reference. In all cases a diagram of the longitudinal and cross-sectional appearance of the fibre is given, but if it were possible within the price actual photographs would have been better.

In the whole book only two types of fibre appear to have been overlooked, viz. chemically crimped viscose rayon and the new Russian 7-nylon. It is regrettable that, with such thorough accounts of the physical properties of the fibres, the book is let down by the casual approach to their chemistry.

This is often very vague indeed, if not misleading. Thus in the case of wool lanthionine is not mentioned as a product of the action of alkali, whilst in a list of the products of oxidation of cystine, cysteic acid is not mentioned at all. In an excellent list of chemical modifications of cotton with acetic anhydride, 2-aminoethyl sulphuric acid, chloroacetic acid, acrylonitrile, etc., not one of the chemical reactions is shown. Whilst the properties of high tenacity viscose fibres are given, no serious attempt is made to show how these fibres are made or why they differ from viscose rayon. The effect of isotacticity is never dealt with, though the properties of polypropylene fibres are well covered.

One gets the impression that the author wishes to avoid any fundamental chemistry of fibres as far as possible, and this, the reviewer feels, detracts considerably from the value of the book.

To sum up one may say that this is an excellent little reference book on the physical properties of all the fibres, but anyone buying it with a view to studying the chemistry of fibres will be disappointed.

R. S. Asquith

Friction in Textiles

By H. G. Howell, K. W. Mieszkis, and D. Tabor. Pp. xii + 263 + V plates. London and Manchester: Butterworths Scientific Publications in association with the Textile Institute. 1959. Price, 42s. 0d.

A comprehensive account of friction and its effects on textile processing is given in this book, which is divided into three main parts: Part I deals with the Theory of Friction and Lubrication, Part II with Friction in Textile Processing, and Part III with Test Methods.

The first section comprises an excellent account of the general mechanism of friction, in which different theories of friction are first discussed. This is followed by an account of an elementary theory of friction which shows how metallic friction may be explained in terms of a simple model. The second chapter of Part I deals with the frictional behaviour of plastics and fibres in which the "point-contact" and "extended line-contact" methods are discussed. An excellent treatment of the mechanism of lubrication forms the final chapter of Part I, though, as the author points out, it is not directly connected with the problems of textile lubrication, although it does include a brief treatment of the general properties of lubricated polymers.

The second part of the book is devoted to friction in textile processing, and covers, among other things, the rôle of friction in the major textile operations of spinning, winding, warping, knitting, and weaving. The first topic dealt with is the friction of wool, which covers the structure of the wool fibre, together with experimental and theoretical studies of the differential frictional effect.

In dealing with friction in spinning, the different theories of drafting are presented extremely briefly, as are the sections on friction in roller drafting and experimental studies of drafting which accompany it. This, together with the shorter chapters which form the second part of the book, is the section which should provide the attraction for the technological reader, yet several aspects of the effect of fibre friction in spinning have been omitted. It is questionable as to whether a textile technologist could derive much immediate benefit from the point of view of yarn production.

An up-to-date account of friction in winding forms the third chapter of Part II, in which is discussed the frictional behaviour of different fibres in yarn form when passing over various types of yarn guides. The effect of lubricants is also discussed. The final chapter of the second section deals with friction in warping, knitting, and weaving, the effect of friction on the strength of tyre-cord, and lastly friction in fabrics.

These last topics have not received a great deal of attention in research as compared with spinning, yet in each case a praiseworthy attempt has been made to present the more important and obvious problems.

The final part of the book is devoted to an excellent and well illustrated review of different experimental techniques which have been used in (i) the measurement of friction of yarns and fibres, and (ii) the control and measurement of tension.

The book is well presented and contains copious references. It represents an extremely good attempt to present an overall and up-to-date picture of the complex topic of friction in textiles and should provide an excellent work of reference for teaching and research.

G. Brook

Alginsäure und Alginate

By Heinrich Maass. Pp. 465. Heidelberg: Strassenbau, Chemie und Technik Verlagsgesellschaft m.b.H. 1959. Price, DM 64.00.

This book is the most complete reference work on alginates that has yet appeared. The author has attempted to cover all that has been published on the production, properties, and applications of alginates and says in his foreword that lack of reference to any source is due to oversight and not rejection. Although he has not fully succeeded in his almost impossible aim, abstracts of some 1,200 papers and patents are given. Some sources published in 1958 are referred to, including several papers given at the Seaweed Symposium held in Galway in August 1958.

Each section of the book begins with a general account of the particular topic, and is followed by a larger part made up of abstracts of the relevant patents and other publications.

The first chapters deal with the occurrence, constitution, and physicochemical properties of alginates. A chapter on the extraction of alginates from seaweed follows, and then sections dealing with alginic acid and its salts, organic compounds of alginic acid and analytical methods. The remaining two-thirds of the book is devoted to the application of alginates and starts with a section on algae as agricultural and industrial raw materials. Publications on the uses of alginate are very well covered, although it is not made clear which of the uses mentioned have in fact developed into successful applications and which have been

found unsatisfactory. This is not the fault of the author, but is a consequence of the nature of the available publications on applications: the majority are patent specifications and many of the others are technical articles written with a sales approach.

Textile applications are covered in two sections, headed *Textiles, Films, and Plastics* (with 39 pp., the longest section in the book) and *Dyes, Printing Pastes, and Paints*. The first of these has alginate yarn as its main topic, but there are some references to textile printing, and it opens with a paragraph on printing pastes. The other section includes detailed print-paste recipes for Indigosol dyes, and among the abstracts are a number dealing with textile printing. There is no mention of the use of alginates in printing with reactive dyes, a growing and important application which has developed in the last few years.

The coverage of patents, taken as a whole, is excellent, but the arrangement of equivalent patents in different countries is confusing. In a few cases the equivalent patents are listed together in one abstract, but as patents are grouped under countries, the corresponding patents are more often distributed among these groups; in some cases all the different patents are abstracted, and in others not. For example, *BP* 555,940 for dissolving calcium alginate with calcium-complexing salts is not mentioned, but the equivalent *Canadian Patent* 466,944 is to be found under "Mixed Salts" and *USP* 2,405,861 under "Jellies".

There is an extensive bibliography and good author and subject indexes. In spite of some criticism of the arrangement of the subject-matter, the book can be recommended as the first work to consult on any question dealing with alginates.

R. H. McDowell

Bleichen und Färben von Textilien

By Walter Bernard. Pp. 240. Berlin: Fachverlag Schiele & Schön G.m.b.H. 1959. Price, DM 18.00.

It will be readily appreciated from the title of this work and from the number of pages which it contains that it can be no more than an introduction. It covers the bleaching and dyeing of cotton, bast, regenerated cellulose, cellulose acetate, man-made, wool, silk, and regenerated protein fibres and still finds space for chapters on the dyeing of unions and the dyeing of textiles. From the first sight of the book one is attracted. The cover is colourful, the paper and printing are excellent, and the illustrations are well chosen and clear. The line diagrams in particular are vivid. As regards the text, the author has succeeded in including something for everybody. There is no great strain placed on the reader's knowledge of chemistry, and, as can only be expected, the treatment is rather two-dimensional. The index shows the wide area covered, however.

Some criticism is called for by the fact that half the book is devoted to cotton bleaching and dyeing. True, much of the matter included in this section is common to the other fibres, but within the section some sense of proportion is lost in equating the importance of hypochlorite, chlorite and peroxide bleaching. There are also more than a few mistakes in addition to those given in the errata. For example, spelling mistakes occur on pp. 133 (Cibocron), 145 (Naphtol AS Pordukte), 147 (Helanka), 168 (Solvay), 170 (Omegrachrom), 181 (Merinowa), 189 (Ommniafarbstoffe), 232 (Montecatine), and 235 (Societé Rapidase). Saran is attributed to Dow on p. 161 and to National Plastics in the fibre table. Tergal and Terylene have exchanged makers on p. 152, whilst on p. 161 the origins of Velon need cleaning up (Fireston, England). The inclusion of Ardil on p. 233 is questionable, and the omission of British Nylon Spinners Ltd. on p. 232 is a serious lapse. The merger of Ciba and Degussa foreshadowed on p. 234 is unlikely to rock the bourses, but requires attention, and Courtaulds is misspelt throughout. The punctuation also calls for careful revision: in common with U.S.A., several Sociétés Anonymes and Aktiengesellschaften, all the stops are pulled out for I.C.I. in the majority of cases and an incorrect address (Milbank) is given.

We can, however, forgive the author; his correspondence file must have been at least as bulky as his MS. He has produced an excellent little book, written with a light touch. There is even humour to be found in the section in which the craft of the dyer is discussed. It should, and doubtless will, be read with pleasure by students, and there is promise of volumes on printing, finishing, fibres, and finishing materials yet to come.

S. R. Cockett

Chemische Technologie und Praxis der Färberei

Band II

Die Farbstoffe und das Färben der Zellulosefasern, synthetischen Fasern und Fasergemische

By Gerhard Nitschke. Pp. 451. Berlin: VEB Verlag Technik. 1958. Price, DM 36.00.

This is the author's second volume under the general heading of *The Chemical Technology and Practice of Dyeing*. Volume I, reviewed in this *Journal* (**71**, 546 (1955)), was concerned solely with wool, but in this instance two broad classes of fibres have been considered together, making the transition to mixtures at a later stage an easier matter.

The work is set out under four main headings: the dyes and dyeing techniques for (1) cellulosic, (2) cellulose ester, and (3) man-made fibres, together with (4) combinations of the three preceding classes. The same regularity of approach which marked Volume I is again apparent with each class of fibre.

Commencing with cellulosic fibres, the direct dyes are considered according to their chemical constitution, followed by the mechanisms of anchoring, of aftertreatments, and the effects of the dyebath variables. A number of special considerations are then dealt with from the practical standpoint. Sulphur, sulphurised vat, vat, leuco-ester, azoic, and basic dyes are all

considered in turn in a similar manner, and finally phthalocyanines and resin-bonded pigment systems are described.

The dyes and dyeing processes for cellulose ester fibres— both secondary acetate and triacetate— are then dealt with including azoic dyes applied by the ethylene glycol–caustic and the Ofna-cet techniques. There follows the section on fully synthetic fibres considered under the headings of polyamide, polyacrylonitrile, polyester, polyvinyl chloride, and copolymers. The author has included the old and the latest techniques, and this section is probably more valuable than the previous chapters. The following application section, which deals with mixtures, is, however, the most outstanding. As pointed out above, the bases have been well laid by the previous treatment of the separate fibres. Unions of wool with silk, with cellulosics, with cellulose acetate, with regenerated protein, and with fully synthetic fibres are dealt with at some length. Cellulosic fibres mixed with silk, with cellulose acetate, and with fully synthetic fibres follow the same pattern, concluding with a few remarks on the dyeing of mixtures containing three or more fibres. It is doubtful whether the dyeing of unions has ever before been considered as systematically as in the present volume, and for this alone the book is worth its price.

Finally, there is an up-to-date section on dyeing machinery, which includes many of the recent innovations and which is well illustrated by figures and plates, frequently in valuable juxtaposition.

The book, apart from the section on unions, is workmanlike but somewhat uninspiring. There is much space wasted with tables of dyes. As far as an author is concerned, these limit the usefulness of a book to the areas where the products are available. From the manufacturers' viewpoint there is free publicity to be had: but it is a duplication of effort, and this type of collation of the products of several companies takes no account of the personalities which colours and speciality chemicals undoubtedly possess. For that reason, possible application without reference to their expertise or published literature is guarded against by manufacturers with Rasputin clauses which, however politely phrased, amount to the same thing. Principles are safer ground for authors: they carry the warranty of Nature.

There are a number of mistakes with foreign names, e.g. "Barator" (p. 395); "Toleries Cantoise" (p. 395); "Standfast Molton Maschine" (p. 406), and— quite inexcusably— half-a-dozen times "C. M. Whittacker".

S. R. COCKETT

Jahrbuch der Textilveredlung
Band 4

By Gerhard Meier. Pp. 818 + 2 folding plates + 1 colour plate. Berlin: VEB Verlag Technik. 1959. Price, DM 40.00.

This volume, being the fourth in its series, indicates a continuing demand for a reference book of this nature in German. Developments in textile chemistry— using the term in its widest sense— are covered for the years 1954–1956, at the same time maintaining a reasonable size. At first sight, comparison with the *Review of Textile Progress* is evoked, but any similarity, apart from that of aim, is rather superficial. It is remarkable that such a small team could have collated all the information contained in the work, but one must conclude that the 200 undertakings and institutions mentioned on the dust-cover must have made significant contributions. The size of the panel, however, must in some measure be responsible for the time-lag in publication.

A major difference from the S.D.C.–T.I. publications lies in the attempt to make the work both a textbook and a reference volume— an attempt which very largely succeeds by the use of excellent systems of arranging the material. This is combined with a standardised treatment under each subheading, which gives a condensation of new information (illustrated with tables, graphs, figures, and plates) followed by a large number of references, the total number of the latter quoted in the work being 6,734. A further compromise which gives the work much wider appeal is the inclusion of technical, technological, and research information as available. The treatment of processing machinery and instrumentation is particularly good.

The general scheme of the book sectionalises the information under the headings of— fibres; general finishing; pretreatments; scouring; dyes, dyeing, and printing; finishing; testing; and laundering, dry cleaning, and garment dyeing. These are then divided and subdivided to give a result which as any essay in marshalling can only be described as exemplary. Although much of the text makes interesting reading, one can assess the true worth of a book of this nature only by making a reasonable number of random appeals. Those carried out as an experiment resulted in much of the required information being forthcoming, or alternatively good leads being given.

It would be surprising, however, if some defects had not crept into an enterprise on this scale. One of the more striking is the difference in levels between sections: but this is surely excusable, since it reflects the differences in the rates of progress in a wide number of fields within a narrow period of time. A minor criticism is the omission of initials in the name index, so that two individuals named Müller and Smith appear to be responsible for a large proportion of the advance in the period covered.

The volume is well produced and will be a useful addition to most libraries alongside the *Review of Textile Progress*, to which it is to some extent complementary.

S. R. COCKETT

The Printing of Textile Fabrics by Block and Screen
A Practical Manual for Use in Colleges and Schools of Art

Fifth edition, 1959. Pp. 159 with 2 fig. Manchester: Imperial Chemical Industries Ltd., Dyestuffs Division. No price.

This is called a fifth edition, but it is, in fact, so different from its predecessors that it should be regarded as a new work. Former editions consisted of two booklets, one of which was a simple

description of the principles of textile printing, its methods, and its styles, and the other was a recipe book, compiled chiefly for schools of art wishing to translate designs into real prints on textile materials. Because the technical training of a number of designers included some practice of weaving, but none of coloration in any real sense, many schools of textile design tended to express themselves chiefly in terms of the woven pattern. This situation is now changing, well equipped installations for screen and block printing are becoming more numerous, and more ambitious ventures are being undertaken.

The present edition is an effort to keep up with these developments. It is now a single and larger volume, and its flavour is almost entirely practical. It is obviously planned to help the beginner to produce successful prints with the chief varieties of dye in most of the styles that a printer might use in putting the work of the designer on to an industrial scale.

Naturally, there is a lot of detail about gear, the printing operations themselves, about steaming, and about the composition of pastes. There are also outline directions for the preparation of different kinds of cloth and for giving simple finishes to the resultant prints. Where industrial procedures cannot be followed on the smaller scale, there are many tips for getting equivalent results by more homely means.

In a treatment of this kind, the amount of space that can be allowed for explanations of how things work and why they are done is very limited. Nevertheless, explanations are interpolated in many places, and prevent the art of printing from looking too much like magic.

In the review of a previous edition, the opinion was given that the colouristic information might be confusingly elaborate for the art-school student, and that it was well beyond what he would need to enable him to convert his designs into realistic prints. This feeling still persists, but its effect as an adverse criticism is minimised by appreciation of the book as a very useful manual for the practical work of students of textile chemistry in universities and technical colleges.

No price is given, and, so far, it is issued as a hand-out, and will go primarily, it is supposed, to teachers, but it is worthy of a wider circulation than could be supported on these terms, and it might be made available for purchase by students and amateurs in general. Two very valuable features deal with, respectively, the preparation and use of silk screens and the construction and operation of small-scale steamers.

In appendixes a great deal of useful information about the composition of solutions, etc., is collected, along with a revised scheme for identifying fibres. An important feature of the last edition is repeated, viz. a list of the suppliers of chemicals and equipment. A most helpful vade-mecum. There are a few errors. Taking it from its context, the most amusing is on p. 14, where, because of undue ellipsis we learn that "a 1 kilowatt heater will liberate 1½ litres (3 lb.) of steam per hour." The art school that could get hold of so much, and such heavy, heavy water, and could superheat it to this extent would be an exciting and dangerous place to work in!

H. A. Turner

Visual Problems of Colour

A Symposium held at the National Physical Laboratory on 23rd–25th September 1957

Symposium No. 8. Volume I. Pp. 1 Plate + viii + 368. Volume II. Pp. ii + 369–749. London: Her Majesty's Stationery Office. 1958. Price, 42s. 0d. (two volumes).

These two volumes reproduce all the papers presented to a symposium held at The National Physical Laboratory in September 1957 together with the ensuing discussions. The forty papers cover almost all aspects of colour vision, both the metrics and the mechanism being considered in detail. A complete list of the papers would be out of place here, but the scope of the symposium can be seen from the subjects discussed at the six sessions— Retinal Chemistry and the Physiology of Vision (Hecht Memorial Lecture), Visual Pigments particularly in relation to Colour Vision, Brightness Matching and Colour Matching, Subjective Colour Measurement and Defective Colour Vision, Electrophysiological Aspects, and Colour Theories. It will be seen from this summary that, taken together, the papers and discussion provide a comprehensive account of the present state of colour science in so far as it relates to the observer's response to the visual stimulus. The papers revealed divergent views on the mechanism of colour vision and showed clearly that we are still a long way from attaining an adequate theory of it. The discussion to which these divergencies gave rise adds considerably to the value and interest of the book.

Naturally, the papers will not all prove of equal interest to colourists, whose main job is to provide the proper visual stimulus. For such readers the papers dealing with colour measurement and discrimination will have the greatest interest. Of particular value in this field is the paper by Dr. W. S. Stiles, in which he presents the results of his new determination of the colour-matching functions of the average observer, which are likely to be embodied in the specification of a new Standard Observer. Outside this field, colour chemists will note the relation between the photochemistry of the visual pigments and that of dyes.

The papers in this symposium have been contributed by workers throughout the world, a noteworthy feature being the large number presented by Russian workers. These volumes are indispensable to anyone interested in the theory of colour vision. They are photoprinted from typescript in a very clear manner, and the price is very reasonable for a production of this size and importance.

F. L. Warburton

The Physical Properties of Polymers

London: Society of Chemical Industry. 1959. Pp. iv + 293. *S.C.I. Monograph* No. 5. Price, 30s. 0d.

This book is a collection of the papers read at the Silver Jubilee Symposium of the Plastics and

Polymers Group of the Society of Chemical Industry, which was held in April 1958. The nineteen papers provide a broad picture of the physical properties of plastics and polymers from both fundamental and technological points of view. Emphasis is on polymers used in the plastics field. For this reason, the first four papers, which deal broadly with structure and properties, are likely to be of most interest to readers of this *Journal*. In the first, C. W. Bunn discusses crystallinity in polymers, its occurrence, measurement, and influence on properties. This paper includes a brief but useful critical review of methods of estimating crystallinity, and the effect of crystallinity on mechanical properties is clearly shown. D. W. Saunders contributes a paper on optical properties of strained amorphous polymers and compares the dependence of stress-optical properties on the type of strain and degree of cross-linking with theoretical predictions. J. Sivadjian describes an interesting method of determining the permeability of polymers to water and water vapour using a photographic technique involving special mercuric iodide and silver iodide plates. P. W. O. Wijga contributes a timely paper on polypropylene in which its structure and properties are discussed.

The remaining papers are of particular interest to the plastics chemist and technologist. Three are concerned with the mechanical properties of plastics in relation to their use and processing. Three more deal with such electrical effects as breakdown of insulation by gaseous discharges, electrical tracking tests, and conduction and polarisation phenomena in plasticised polymers. Two more reflect current interest in toughened polymers, considering tests on impact strength and the variation of the physical properties of melamine moulding materials with curing time respectively. Four papers deal with such aspects of elastic effects as rebound resistance, dynamic elastic measurements, viscoelastic effects in ethylene polymers, and dynamic properties of polyvinyl chloride. The final three papers are concerned with dynamic mechanical behaviour. One considers partially crystalline polymers. The others are concerned with the influence of strain on the dynamic properties of rubber and the mechanical and dielectric temperature/frequency equivalence of a synthetic rubber.

There are many useful tables and clear diagrams in the book, not the least interesting parts of which are the discussions following each paper. Although it provides details of certain specialised aspects rather than a general account of physical properties and is essentially a book for the plastics chemist or technologist, there is much of interest to the chemist concerned with polymers generally.

W. R. Moore

New Books Received

Printing Ink Technology. E. A. Apps. London: Leonard Hill (Books) Ltd. 1958. Pp. xxii + 552 + 11 plates. 70s. 0d.

Principles of Biological Microtechnique. A Study of Fixation and Dyeing. John R. Baker. London: Methuen & Co. Ltd. (New York: John Wiley & Sons Inc.). 1958. Pp. 357 + 9 half-tones. 45s. 0d.

The Emergence of the German Dye Industry. John Joseph Beer. Urbana, Illinois: University of Illinois Press. 1959. Pp. vii + 168. Paper $3.50, cloth $4.50.

Bleichen und Färben von Textilien. Walter Bernard. Berlin: Fachverlag Schiele & Schön G.m.b.H. 1959. Pp. 240. DM 18.00.

British Standards Yearbook 1959. London: British Standards Institution. [1959.] Pp. iii + 543. 15s. 0d.

Improved Inks for Letterpress Four-colour Printing. B.S. 3020 : 1959. London: British Standards Institution. Pp. 11 + 5 colour plates. 6s. 0d.

Amendment No. 1, published 26 June 1959 to British Standards Handbook No. 11 : 1956. Methods of Test for Textiles. PD 3439. London: British Standards Institution. Pp. 9.

Handbook of Textile Fibres. J. Gordon Cook. Watford: Merrow Publishing Co. Ltd. 1959. Pp. xxv + 422. 15s. 0d.

Věda a Výzkum v Průmyslu Textilním. Sborník Přednášek z II Koloristického Sjezdu v Tatranské Lomnici 1957. Řada IV–1958. Prague: Czechoslovakia Ministerstvo Spotřebního Průmyslu. Pp. 155.

Hydrogen Bonding. Papers presented at the Symposium on Hydrogen Bonding held at Ljubljana, 29th July–3rd Aug. 1957. D. Hadži (editor) with the co-operation of H. W. Thompson. London: Symposium Publications Division, Pergamon Press Ltd. 1959. Pp. xii + 571. 110s. 0d. ($17.50).

Textile Recorder Annual 1959–1960. Manchester and London: Harlequin Press (1955) Ltd. [1959.] Pp. 154 (including adverts.). 30s. 0d.

The Printing of Textile Fabrics by Block and Screen. A practical manual for use in colleges and schools of art. Manchester: Imperial Chemical Industries Ltd., Dyestuffs Division. 5th edition 1959. Pp. 158. No price.

Notes for Authors. A guide for contributors to the Institute's journals and other publications. London: Institute of Physics. 1959. Pp. 36. 3s. 6d.

Centuries of Achievement in Wool. London: International Wool Secretariat, Department of Education. 1959. Pp. ii + 88. 3s. 6d.

Alginsäure und Alginate. Heinrich Maass. Heidelberg: Strassenbau, Chemie und Technik Verlagsgesellschaft m.b.H. 1959. Pp. 465. DM 64.00.

Jahrbuch der Textilveredlung. Band **4**. Gerhard Meier. Berlin: VEB Verlag Technik. 1959. Pp. 818 + 2 folding plates + 1 colour plate. DM 40.00.

Methoden der Organische Chemie (Houben-Weyl). Band **I**, Teil 2: *Allgemeine Laboratoriumspraxis II.* E. Müller (editor). Stuttgart: Georg Thieme Verlag. 4th edition 1959. Pp. xlviii + 1017 + frontispiece + 2 folding tables. DM 196.00.

Symposium on Colour Tolerance, London 2nd April 1958. Physical Society Colour Group. Göttingen, Germany: Musterschmidt-Verlag. 1958. Pp. 193–255. 30s. 0d. (In English.)

Influence of Colorant Systems on Thermal Protection. Parts III, IV, and V. Alvin O. Ramsley. Natick, Massachusetts: United States Army, Headquarters, Quartermaster Research and Engineering Centre, Textile Clothing and Footwear Division. Feb. 1959. Pp. viii + 52 + vi. No price.

The Physical Properties of Polymers. Monograph No. 5. London: Society of Chemical Industry. 1959. Pp. iv + 293. 30s. 0d.

Der Rakeltiefdruck. Aus der Praxis eines Tiefdruck Technikers. Adalbert Strakaty. Mauthausen, Austria: Verlag der Rakeltiefdruck A. Strakaty. 10th edition 1959. Pp. 116 + 1 colour plate.

[*Russian–English–Chinese Glossary of Organic Dye Terms*]. [via Collet's Scientific Bookshop.] 1956. Pp. ii + 233. 15s. 0d.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Hard Chromium Plating of Printing Rollers

G. Monte *Melliand Textilber.*, **40**, 184–188 (Feb. 1959)

Review of historical development, suitable current densities (20–50 amp./sq.dm.), temp. (35–55°c.), and voltages (6–8 v.). The advantages of chromium plating are discussed. Secondary electrolytic processes reduce current yields to ca. 20%. Maximum efficiencies require the presence of acids not derived from Cr in amounts constituting 1% of the CrO_3 content. H_2SO_4 is preferred to the more efficient HF, because of the aggressive action of the latter on anodes and workers. 300–400 g. per litre CrO_3 are preferred. The author then deals with current density–temp. relationships, analytical control, pretreatment of rollers, and hygiene. Some useful references are embedded in the text. S.M.J.

PATENTS

Carbon Black Furnace

Commercial Solvents Corpn. *USP* 2,885,269

Apparatus for the continuous production of Carbon Black (C.I. Pigment Black 6 and 7) from cheap hydrocarbons. C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)

Esso Research & Engineering Co. *USP* 2,885,267

Apparatus for the cracking of methane or other hydrocarbon gas to produce high purity hydrogen and Carbon Black. C.O.C.

Carbon Black (C.I. Pigment Black 6 and 7)

Godfrey L. Cabot *BP* 819,823

A process for recovering the pigment from effluent gas by shock cooling in a manner which does not involve addition of water vapour. C.O.C.

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. *USP* 2,883,274

Method and apparatus for converting the light, flocculent pigment into free-flowing, dustless, small pellets. C.O.C.

Recycling Carbon Black (C.I. Pigment Black 6 and 7) Pellets during Pelleting

Phillips Petroleum Co. *USP* 2,878,511

Automatic Control of the Continuous Application of Specified Amounts of Solids or Liquids to a Moving Sheet

British Cotton Industry Research Assocn. *BP* 817,838

Method for avoiding the necessity for a reserve of stock liquor slurry in a machine of the type described in *BP* 654,178 and 792,663 (J.S.D.C., **67**, 347 (1951); **74**, 565 (1958)). C.O.C.

Drying Travelling Textile Threads

J. P. Bemberg *BP* 819,997

The threads are led over a drum rotating at the speed of travel of the threads. This drum has a large number of juxtaposed electric contacts, adjacent contacts being charged with voltage of different polarity. By this means there is no relative movement between contacts and threads, so that a firm connection is formed between both parts thus permitting satisfactory and non-damaging flow of current. C.O.C.

Steam-heated Rollers

VEB Spinn- und Zwirnereimaschinenbau Karl-Marx-Stadt *BP* 819,808

A steam-heated roller specially suitable for heating rayon filaments during their manufacture has an annular space for the passage of steam. This space is bound by the outer wall of the roller and cylinder placed concentrically within the roller. This cylinder is axially displaceable to compensate for expansion. C.O.C.

Hank Dyeing Machine

Samuel Pegg & Son *BP* 816,467

A cage in which the hanks are suspended can be lifted into and out of a hollow casing having a removable lid. The casing is divided into two compartments by a partition between its end walls. Both the compartments are filled with hanks suspended from poles. A pump circulates the dye liquor through the two compartments in turn. Static liquid pressure can be applied to the dye liquor. The machine can be used for high temperature dyeing. C.O.C.

Weft Straightener

J. D. Robertson *BP* 816,491

Fabric Dyeing Machine

B. Thies K.G. Specialmaschinenfabrik *BP* 816,546

A high temperature (< 140°c.) machine in which the cloth is suspended above the liquor contained in the bottom of a closed vessel. The liquor is drawn from the bottom of the vessel by a pump and sprayed down on to the cloth. C.O.C.

Treating Cloth with Gas, Vapour or Liquid

Naamlooze Vennootschap P.F. van Vlissingen & Co.'s Katoenfabrienken *BP* 816,703

Modification of *BP* 694,158 (J.S.D.C., **69**, 382 (1953)). The cloth passes over upper and under lower rollers. The lower rollers freely rotate on fixed axes whereas the upper rollers are cylindrical and driven so that their relative peripheral speeds decrease or increase successively from the inlet to the outlet of the machine. C.O.C.

Composite Clip and Pin Selvedge Holders for Tenters

John Dalglish & Sons *BP* 817,047

Web Tensioning Device

E. A. Timson *BP* 816,859

Simple and effective means for ensuring constant tension in a web, particularly one of thin and flimsy material, while it is being printed, embossed or subjected to similar treatment. C.O.C.

Embossing Rolls

Eastern Engraving & Machine Co. *USP* 2,887,042

Method of engraving a female steel roll and mating it at optimum pitch angle to a matching male steel roll. C.O.C.

Facilitating the Introduction or Removal of Cylinders into Printing, Embossing or Waxing Machines

James Halley & Sons *BP* 819,917

Raising Machine

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A machine giving more precise control over the cloth. The angle of inclination of the teazles of each row being adjustable there is less restriction on the amount of raise than in previous machines. C.O.C.

Raising Machine

Riggs & Lombard *USP* 2,884,678

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Fabric Press

Edwin Mills & Son *BP* 817,282

The cuttled pieces are pressed, there being a board beneath and on top of the stack of pieces. There are connecting members which engage the two boards when the pieces have been compressed, these connecting members being readily released after the stack of pieces has been removed from the press. C.O.C.

Drying Coated Webs

J. H. Flynn *USP* 2,884,705

A method of drying coated webs by use of an open flame. Speeds of 2,000 ft. per min. or more are claimed. C.O.C.

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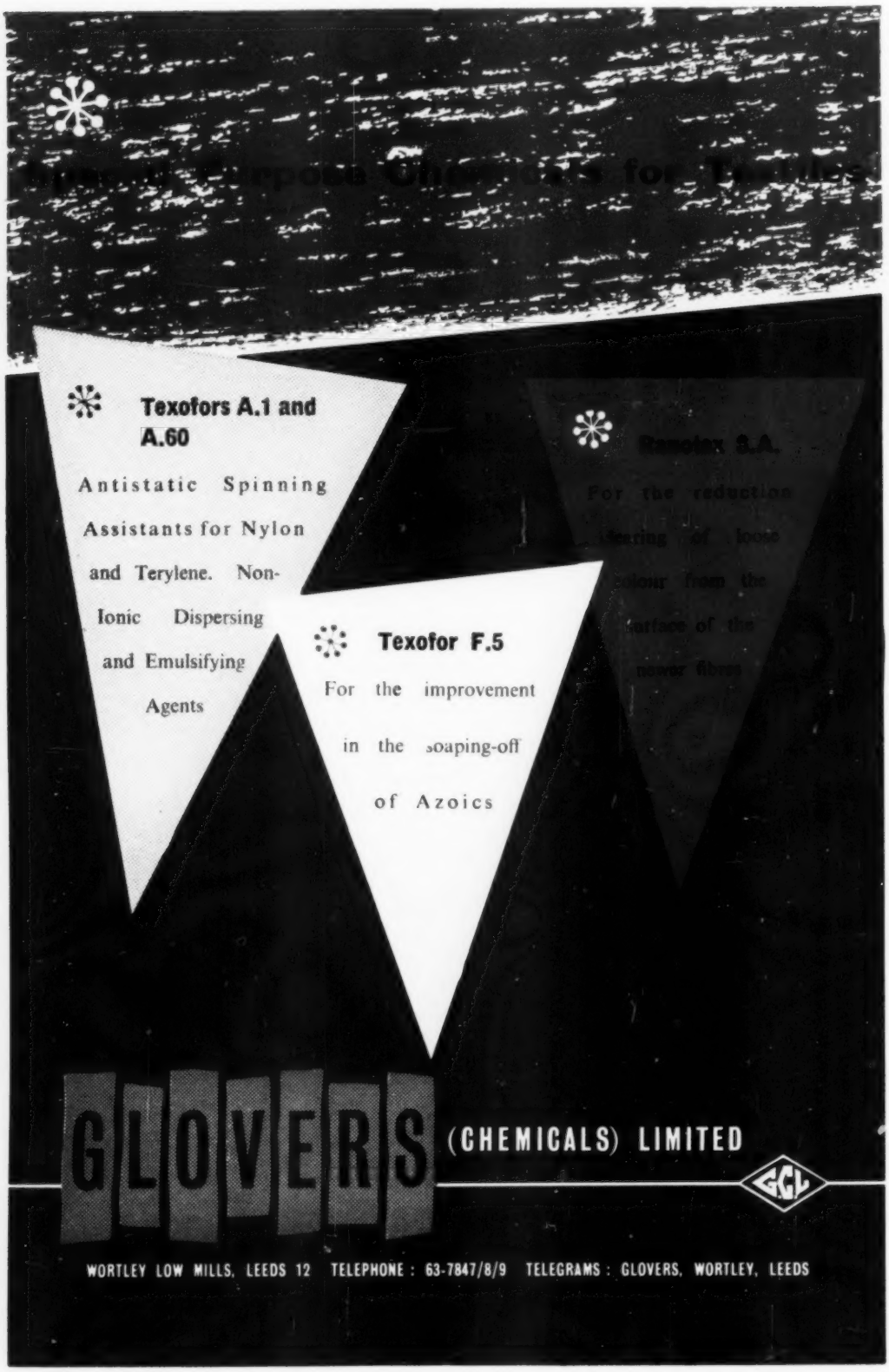
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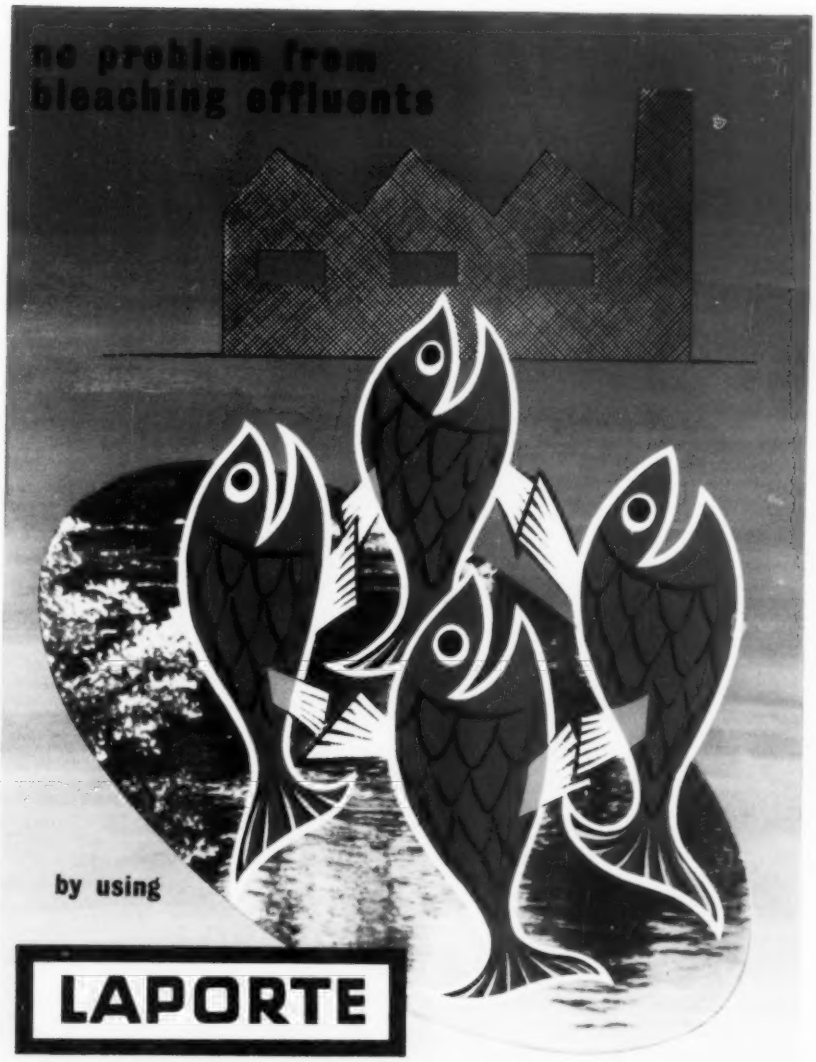

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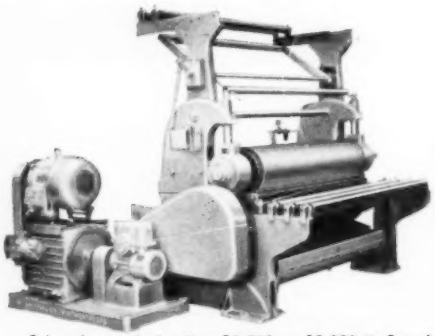

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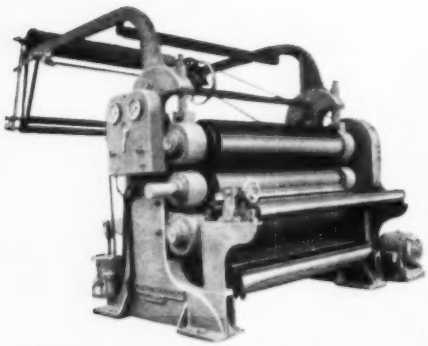

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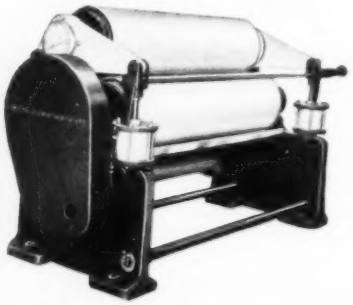

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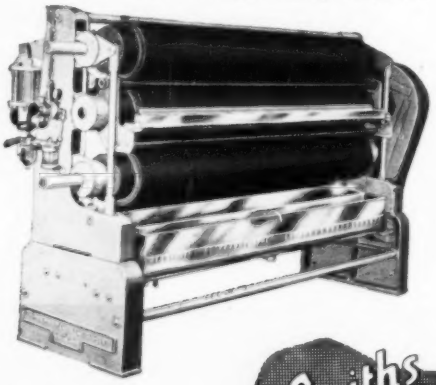

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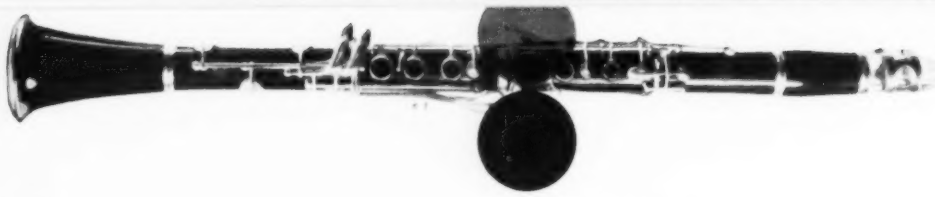

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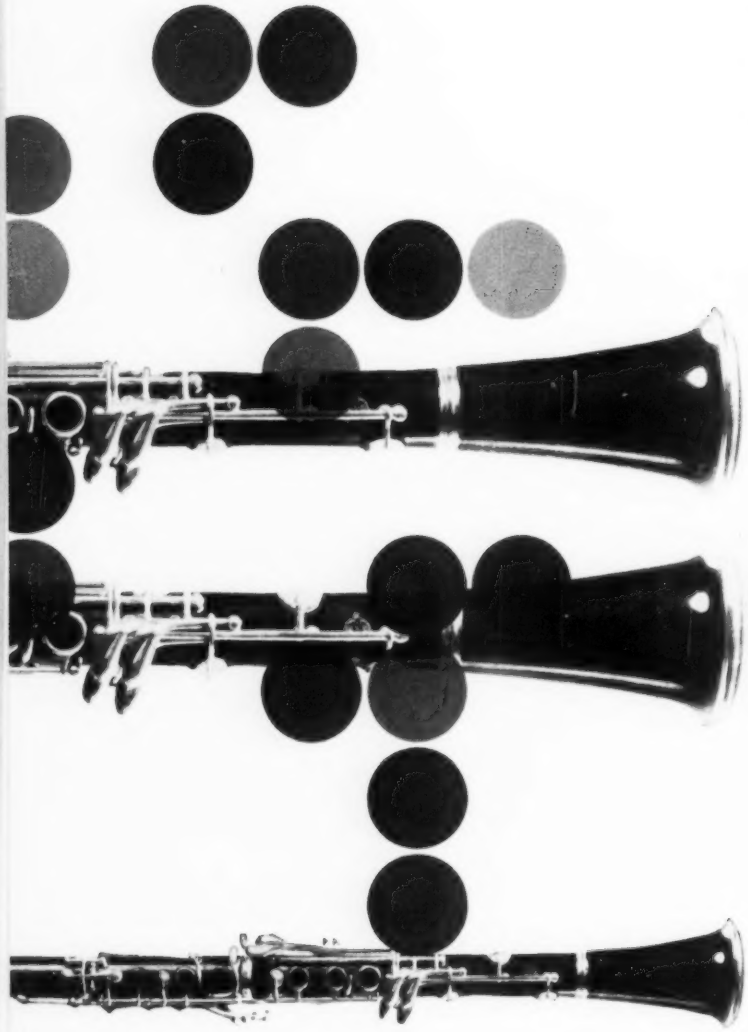

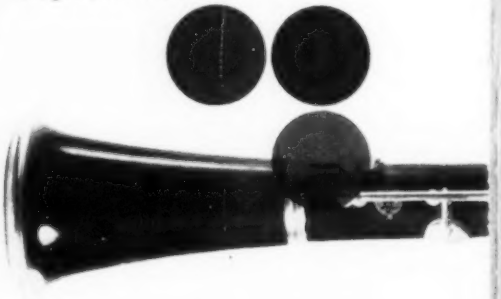

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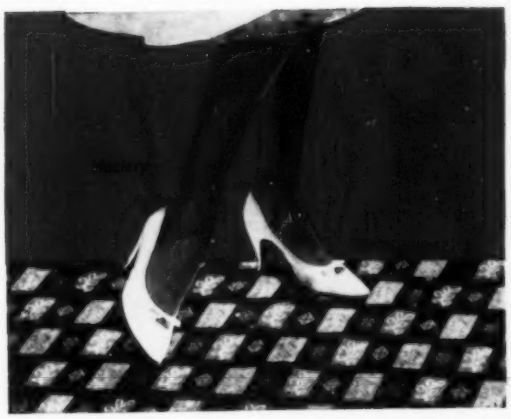

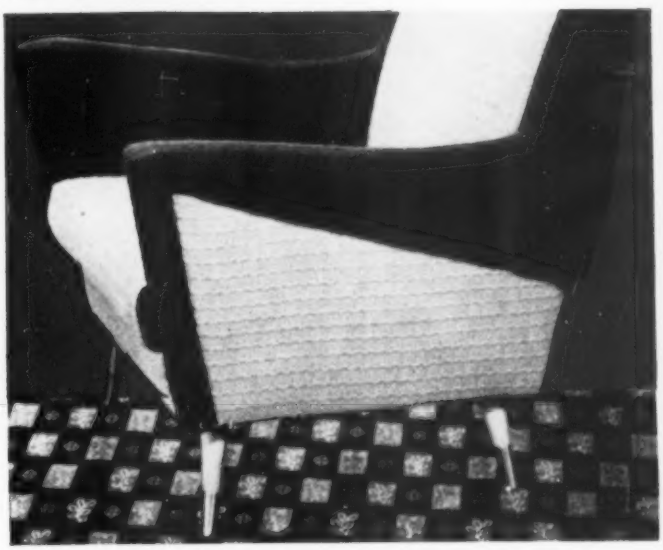

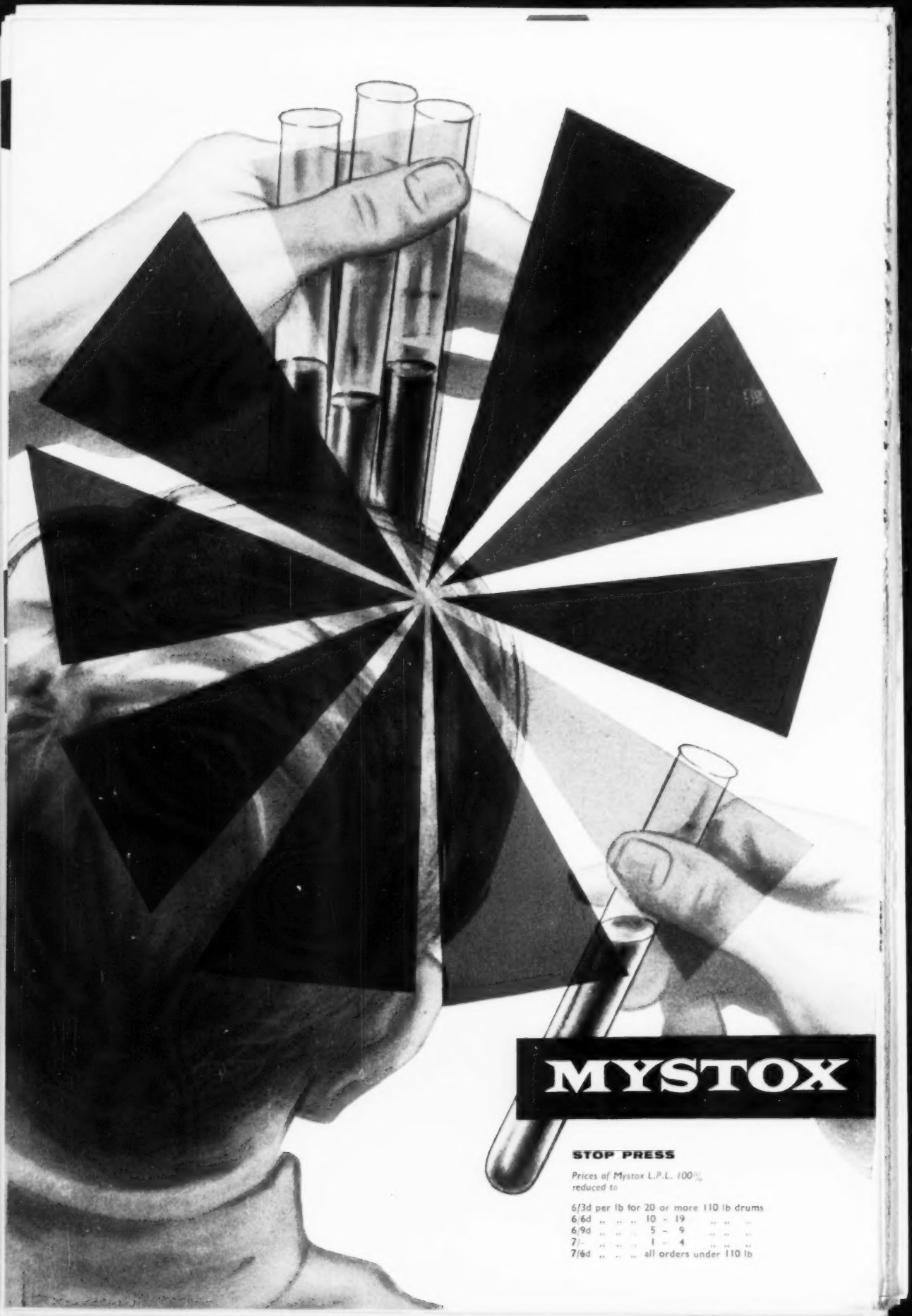

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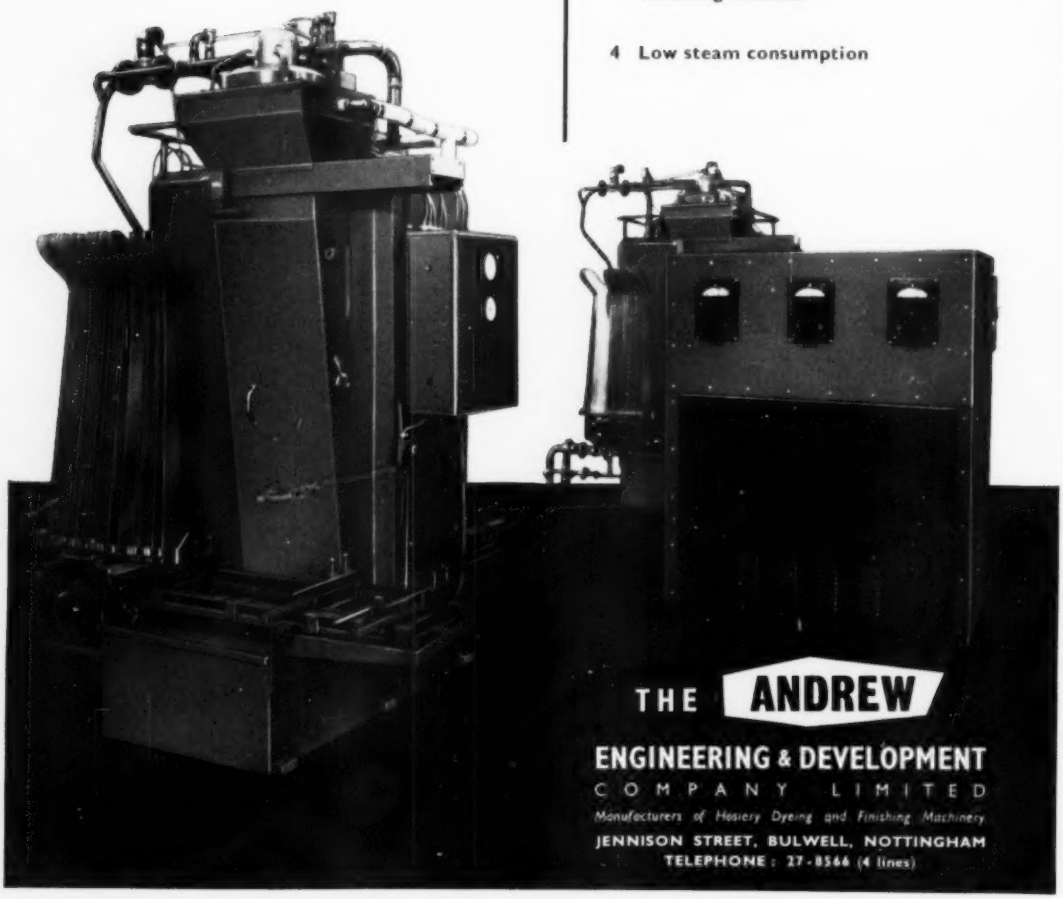

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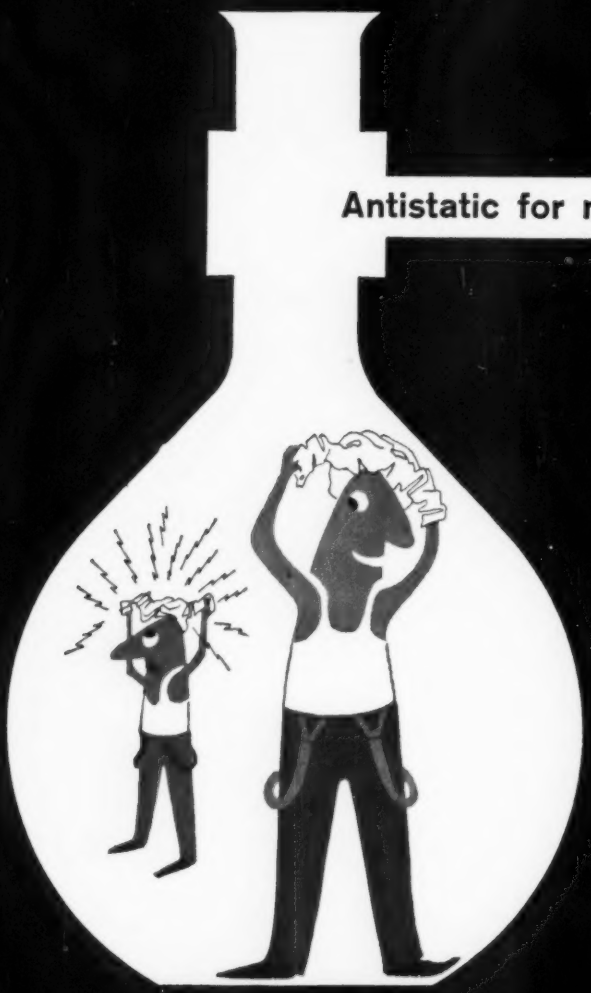

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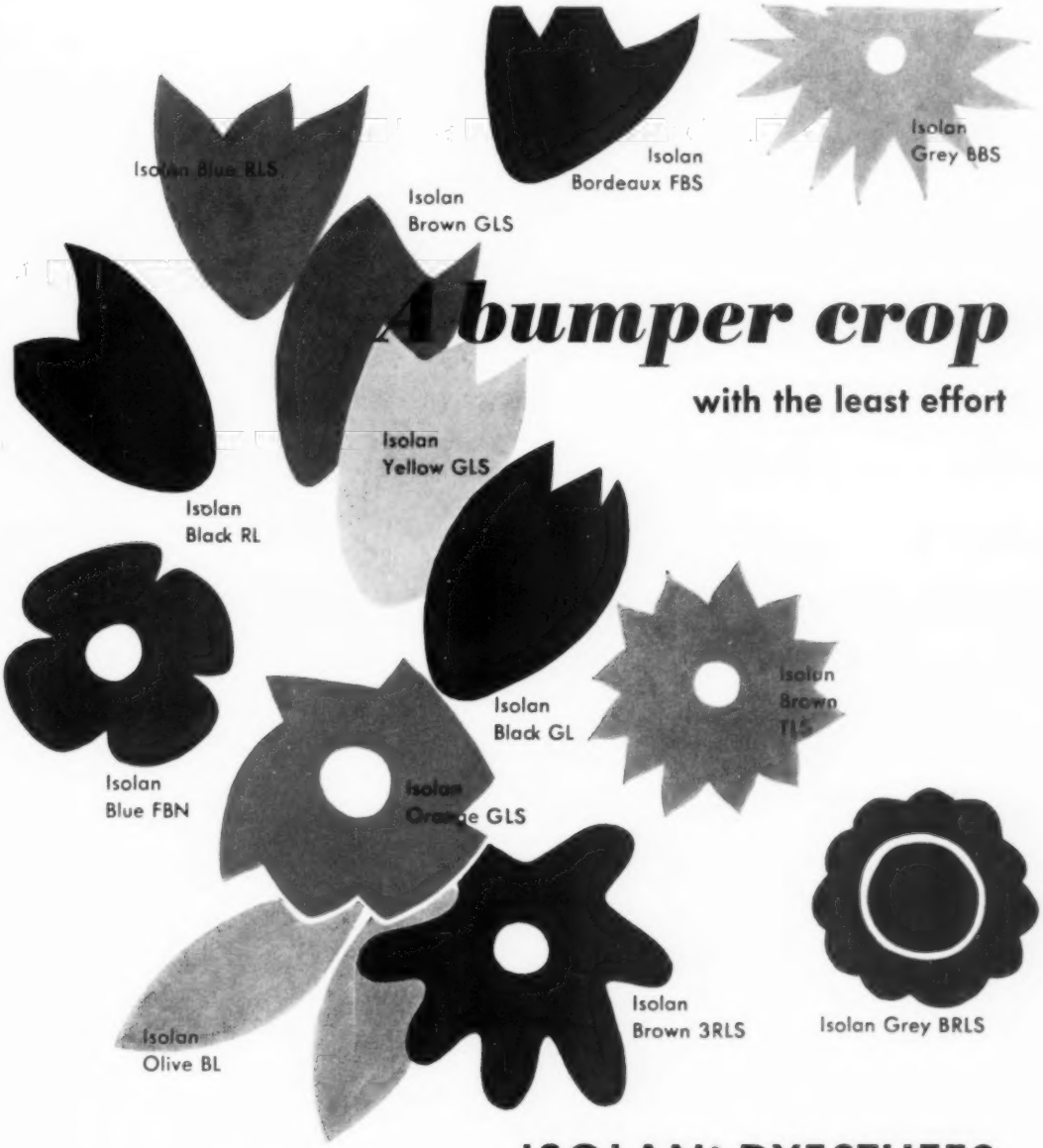

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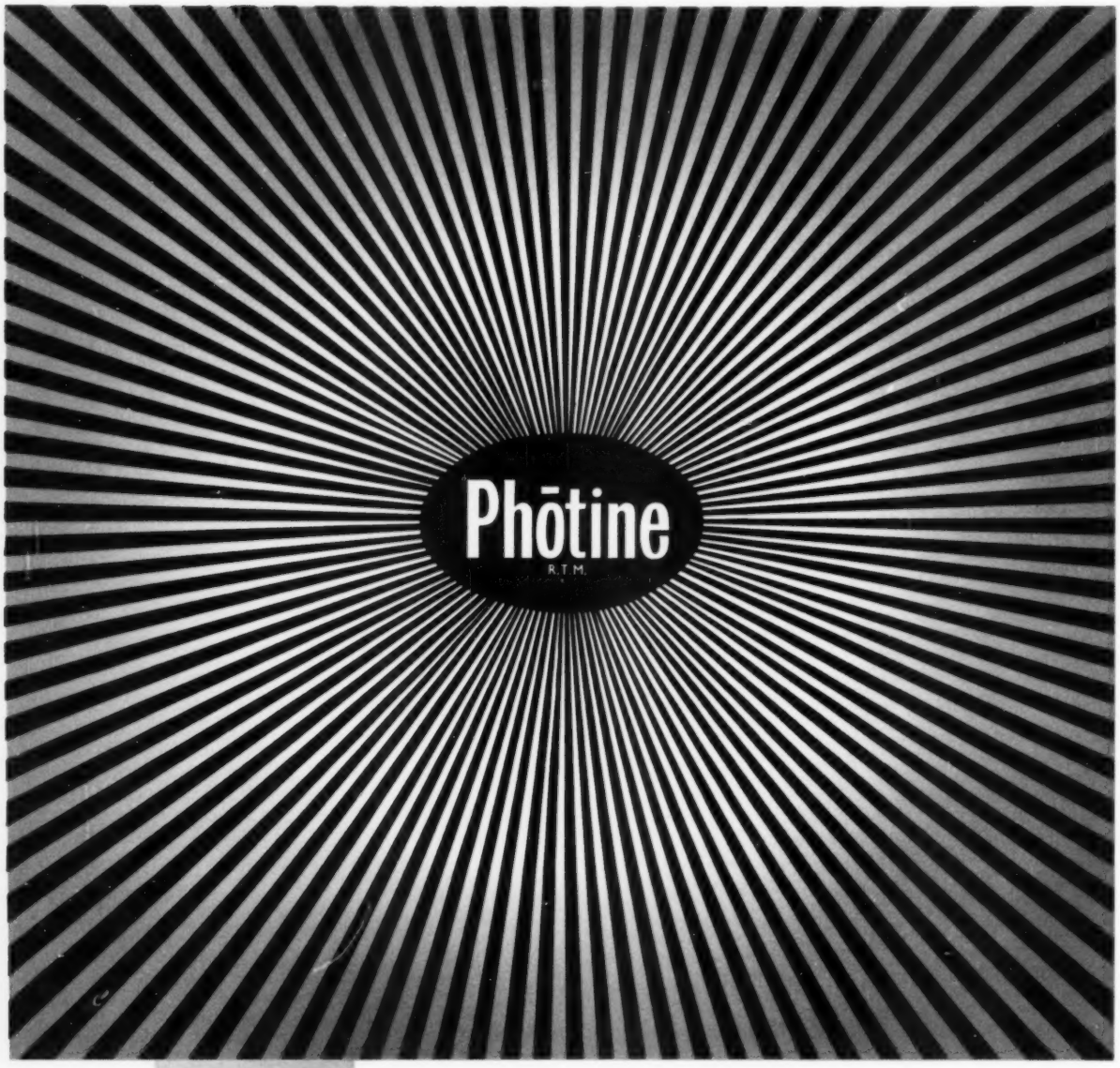

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Velveray Corpn. *USP* 2,881,087
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Dexdale Hosiery Mills *BP* 816,130
Apparatus which eliminates separate preboarding and finish boarding of nylon stockings or the like. The goods are mounted on forms and placed in a closed chamber where they are treated with hot air before being steamed and then dried with hot air before being removed from the chamber. A control enables the preliminary heating with hot air to be cut out whenever necessary. C.O.C.

Machine for Shrinking and Felting Hat Bodies
John B. Stetson Co. *BP* 816,494

Monitoring and Control Apparatus for Use in Multicolour Printing
J. F. Crosfield *BP* 820,361

Treating Tubular Knit Fabric with Resins (X p. 574)
Laminated Sheet Material (X p. 575)
Applying Flock to the Surfaces of Irregularly-shaped Articles (XIII p. 577)

II—WATER AND EFFLUENTS

Boiler Scale Prevention
R. Freitag *Melliand Textilber.*, **40**, 213–215 (Feb. 1959)
Supersonic methods of preventing scale deposition by changing the crystal structure of $CaCO_3$ are illustrated. S.M.J.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Aliphatic Surface-active Agents: Development and Raw Materials
H. Machemer *Melliand Textilber.*, **40**, 56–65 (Jan.), 174–179 (Feb. 1959)
An extensive review of the whole field from its earliest beginnings. A summary is followed by discussion of surface-active agents based on oils and fats, such as Turkey Red Oil, alkylnaphthalenesulphonic acids, sulphonated fatty acid amides and alkyl esters, alkyl sulphates, Igepons (IG), and Ultravons (Ciba). Agents on a fat free basis are then considered, with reference to the Fischer–Tropsch process, sulphochlorination, paraffin oxidation, non-ionics, the Oxo process, Teepol, alkyl aryl sulphonates, and cation-active agents. Syntheses of higher alcohols by condensation of aldehydes and ketones, and of lower alcohols, and catalytic hydration of oxides of carbon are then described. The author deals further with the effect of chain branching on surface activity of C_8–C_{16} alcohols and alkyl sulphates, emphasising the efficiency of α-branched hydrophobic groups with polyatomic substituents. Details of product composition of many syntheses are provided. Numerous tables and graphs illustrate viscosities, surface tensions, wetting and foaming power. There is an exceptionally large number of patent and literature references, occupying 12 columns. S.M.J.

Organosilicon Compounds for Water-repellent Finishing of Cotton
N. V. Kalugin and M. G. Voronkov
Khim. i Prakt. Primenenie Kremneorg. Soedinenii, Trudy Konf., Leningrad, (4), 54–64 (1958): *Chem. Abs.*, **53**, 11848 (25 June 1959)
Tests on tent cloths were made with an emulsion of $(C_2H_5SiHO)_n$ (I) and solutions of $CH_3Si(OCOCH_3)_2$·$OCOC_{17}H_{35}$ (II), $[(CH_3)_2SiOSi(OCOCH_3)_2OSi(CH_3)_2O]_n$ (III), and $CH_3Si(OH)ONa$ (IV). Preliminary tests showed that cloths treated with (III) soon lost their hydrophobic properties, so further trials with this compound were abandoned. The other three compounds were applied immediately after the test samples had been given the standard Russian rotproofing treatment for tent cloths (use of an oak extract with Cu and Cr salts). Much difficulty was met with in the application of (II) because of the organic solvent necessary but several methods were worked out using $Ti(OC_2H_5)_4$ as catalyst. Trials lasting 5 months showed that I, II, and IV can all be successfully used in conjunction with the standard Russian rotproofing process. The deterioration caused to the fibres by the silicone treatment is adequately compensated for by the durability of the water-repellent finish. Their use with the Cu and Cr salts of the rotproof process ensures durability of the finish. It is recommended that this method replace the present practice of using Al salts. C.O.C.

PATENTS

Detergents containing Perborates and a Copper Salt
Food Machinery & Chemical Corpn. *BP* 819,842
Addition of a water-soluble copper salt to a mixture of an alkyl aryl sulphonate and a perborate yields a mixture which has good detergent and bleaching action even when kept for prolonged periods, e.g. a mixture of alkyl aryl sulphonate (140 by wt.), Na tripolyphosphate (140), tetrasodium pyrophosphate (60), carboxymethyl cellulose (4), sodium metasilicate (22·8), sodium perborate tetrahydrate (64), and $CuSO_4 \cdot 12H_2O$ (2·84). C.O.C.

Wetting Agent having Low Tendency to Foam in Aqueous Solution
DH *BP* 815,858
A mixture of a water-soluble salt of *NN*-dibenzylaminobenzene 3- or 4-sulphonic acid and a water-insoluble neutral triester of phosphoric acid (15–70% by wt. on the weight of the free sulphonic acid), e.g. tri-*n*-butoxyethyl phosphate, has excellent wetting power and little tendency to foam in aqueous solution. C.O.C.

Surfactants derived from Acrylates and Methacrylates
Shawinigan Chemicals *USP* 2,883,369
Highly effective surfactants are obtained by treating acrylic acid, methacrylic acid, acrylonitrile, methacrylonitrile or their alkyl esters (Alk of < 11 C) with SO_3 and a hydroperoxide, e.g. t-butyl or cumene hydroperoxide. The products contain SO_3H groups and are strongly acidic. They are effective oil-soluble emulsifying and stabilising agents particularly for vinyl acetate and acrylic esters. Such dispersions yield coatings which after being dried have excellent clarity, gloss and resistance to water. The alkali metal salts of these acids are also very effective emulsifying and stabilising agents. C.O.C.

Polyfunctional Surface-active Agents
Société Anonyme d'Innovations Chimiques dite: "Sinnova" ou "Sadic" *BP* 816,888
Treating degraded protein with a halogenated acid chloride and then condensing with an amine yields surface-active agents which have effective wetting and detergent properties in hard or soft water and under both acid and alkaline conditions. C.O.C.

Emulsifying Agents
Atlas Powder Co. *BP* 816,915
A mixture of a polyoxyethylene ether of a hexitan monoester of tall oil (10–20 oxyethylene groups per mol.) and a polyamine salt of an alkyl aryl sulphonic acid is an excellent emulsifying agent for producing aqueous emulsions of hydrocarbon solvent solutions even with hard water. C.O.C.

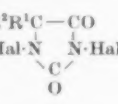
Epoxy-methylol Phosphorus Derivatives
Albright & Wilson *BP* 816,069
Production of polymers similar in type and uses to those of *BP* 740,269; 761,985; 764,313/4 and 807,236 (J.S.D.C., **72**, 40 (1956); **73**, 61, 216 (1957); **75**, 221 (April 1959)) is described. C.O.C.

Bleaching and Disinfecting Compositions
Purex Corpn. *BP* 816,882
A mixture of di- or trichlorocyanuric acid with a salt complex obtained by drying an aqueous slurry of an alkali metal silicate and an alkali metal sulphate, carbonate or chloride is used as a bleaching and disinfecting composition. The salt complex prevents the chlorocyanuric acid from giving off lachrymatory odours. C.O.C.

Colloidal Silica for Improving the Spinning Properties of Fibres
Monsanto Chemical Co. *USP* 2,885,308
Treatment with silica of particle size 250–800 mμ. results in marked decrease in slippage of fibres during spinning and in yarn of very high tensile strength. C.O.C.

Hydantoin-modified Starch
H. Robinette *USP* 2,880,107

The micellar structure of starch is modified by treating it with a compound of formula—



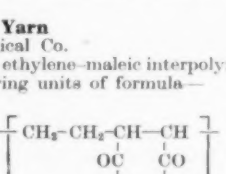
(R^1 and R^2 = same or different, H or non-functional radical), e.g. *NN*-dichloro-5-methylhydantoin. The treated starch produces better films and has improved penetrating properties. C.O.C.

Starch–Bisulphite Adducts
U.S. Secretary of Agriculture *USP* 2,880,236

Dialdehyde starch, i.e. starch which has been oxidised with a periodate, when treated with an aqueous bisulphite or H_2SO_3, forms adducts which can be obtained as white solids. These solids are stable when stored at ordinary temperatures and are readily soluble in water for use as sizes, adhesives, and thickening agents for textile printing pastes. C.O.C.

Size for Nylon Yarn
Monsanto Chemical Co. *BP* 815,830

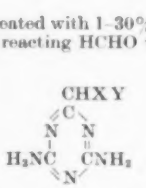
Water-soluble ethylene–maleic interpolymers containing numerous recurring units of formula—



(one X = H and the other = H or is derived from an alcohol X·OH) and of viscosity $\leqslant$ 0·1 in a 1% by wt. solution in dimethylformamide at 25°c. are good non-tacky sizes for nylon even under severe conditions of humidity. They must be used as the free acids the alkali salts being greatly inferior as sizes. C.O.C.

Guanamine–Formaldehyde Condensates for Treating Textiles
W. J. van Loo *USP* 2,887,409

Cellulosic fabrics treated with 1–30% of their weight of a product obtained by reacting HCHO with a compound of formula—



(X = Alk of 1–4 C, alkoxy of $<$ 9 C and alkoxy alkoxy of $<$ 9 C; when X = Alk then Y = OH; in other cases Y = H), e.g. methoxyacetoguanamine, and then cured. The mole ratio of HCHO to guanamine is $\leqslant$ 3:1. The treated fabrics have good crease recovery and are not susceptible to loss of strength or discoloration when bleached with chlorine. C.O.C.

Dextran Solutions as Finishes for Paper and Textiles and for Making into Fibres
Commonwealth Engineering Co. of Ohio *BP* 815,863

Dextran of mol.wt. 20,000–200,000 dissolves in aq. 50–70% BF_3 at $>$ 30°c. and $<$ −5°c. The dextran may be reprecipitated from the solution either by dilution with water or treating with alkali. C.O.C.

Size containing Dextran
Commonwealth Engineering Co. *USP* 2,880,105

Size comprising dextran and an aldehydo quaternary ammonium salt, e.g. ethoxymethyl dimethyl β-methyl-β-formyl-propyl ammonium chloride, $C_2H_5{-}OCH_2{\cdot}N(CH_3)_2Cl{\cdot}CH_2C(CH_3){\cdot}CHO$, is insolubilised when heated *in situ* on the fibre. C.O.C.

Tall Oil Rosin Size stabilised against Crystallisation
Hercules Powder Co. *USP* 2,881,084

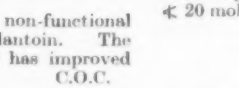
Crystallisation in paste tall oil rosin sizes by adding 5–20% on the weight of rosin of a rosin acid ester of ethylene glycol, pentaerythritol or sorbitol. C.O.C.

Lead Alloys for Coating and Sizing Glass Fibres
B. F. Goodrich Co. *BP* 820,276

Alloys of 0·001–10·0% of Pb with Na, K, Mg, Ca, Zn, Sr, Ba, Al, Ga, Ti, Sn, Co, Mn, Cd or Fe readily wet glass fibres. C.O.C.

Quaternary Ammonium Compounds of Polymers of Aminoalkyl Vinyl Ethers— Auxiliary Agents
Rohm & Haas Co. *BP* 815,745

The quaternary ammonium compounds of polymers of monoethylenically unsaturated monomers containing $\leqslant$ 20 mol. % of units of formula—



(A = ethylene, trimethylene or $-CHR^4{\cdot}CH_2-$ (R^4 = Alk or alkenyl of 1–16 C); R^1 and R^2 = (1) same or different, Alk or alkenyl of 1–12 C, hydroxalkyl of 2–12 C; alkoxyalkyl of 2–12 C, (polyalkoxy)alkyl of 2–12 C, or (2) together = morpholino, pyrrolidino, piperidino or *N*-alkylpiperazino :$(C_2H_5)_2{:}NR^5$ (R^5 = Alk of 1–18 C); R^3 = satd. and ethylenically unsatd. aliphatic hydrocarbons of 1–18 C (polyalkoxy)alkyl of 2–18 C, alkoxyalkyl of 2–18 C, hydroxyalkyl of 2–18 C, aralkyl or subst. aralkyl of 7–24 C, or a phenoxy alkyl of 7–24 C; at least one of R^1, R^2, and R^3 has $>$ 11 C; X = OH or a negative salt-forming atom or radical) have an extremely wide range of uses, e.g. rot proofing agents, antistatic agents, softening agents, improving the dyeing properties of cellulose acetate, etc. C.O.C.

Antistatic Agent
Dow Chemical Co. *BP* 815,947

Copolymers of a sulphonated styrene monomer and a vinyl pyridine compound, e.g. a *p*-styrene sulphonic acid-2-vinyl pyridine copolymer, are excellent antistatic agents particularly for polymers containing acrylonitrile. C.O.C.

Melamine–Formaldehyde Condensates— Antistatic Agents
Ciba *BP* 816,044

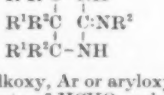
The condensate (1 mol.) of (1) a formaldehyde condensation product of melamine or an ether thereof with an alcohol of 1–4 C and (2) a compound containing a basic testing N atom which is a hydroxy ethylated aliphatic amine or a hydroxyethylated polyethylene polyamino of 3–5 N acylated at a N atom by a fatty acid of 12–18 C or a hydroxyethylated piperazine, is quaternated with $>$ 1 mol. epichlorohydrin in absence of water. Thus dioxyethylpiperazine (obtained by treating piperazine with ethylene oxide at 130–140°c.) when condensed with hexamethylol-melamine hexamethyl ether and then treated with epichlorohydrin yields a water-soluble product which can be used as an antistatic agent. C.O.C.

Antistatic Agent
Agfa *BP* 819,945

Acetals derived from a linear hydroxylic polymer and an alkali metal salt of a sulphonated aromatic aldehyde are antistatic agents specially suitable for use with photographic films, e.g. an acetal of poly(vinyl alcohol) (mol.wt. 5000) and Na benzaldehyde 2,4-disulphonate. C.O.C.

Resin Finishes
Proctor Chemical Co. *USP* 2,884,301

The condensate of an aldehyde and a heterocyclic compound of formula—



(R^1 = H, OH, Alk, alkoxy, Ar or aryloxy; R^2 = H, Alk or Ar), e.g. the condensate of HCHO and tetrahydro-2(1H)-imino pyrimidine, is used to produce a crease shedding finish which has no chlorine retentive or odour-forming properties. C.O.C.

Phosphorus–Nitrogenous Polymers for Rendering Textiles Flame Resistant
U.S. Secretary of Agriculture *USP* 2,886,538

The polymers produced by reacting a 1-aziridinyl phosphine oxide or sulphide with a polyhydric alcohol of

formula $R(OH)_x$ ($x = > 1$; R = Alk or Ar) are useful flame-resistant agents. Thus pentaerythritol (25) is dissolved in water (200) at 30–35°C. and then tris(1-aziridinyl)phosphine sulphide (25) is also dissolved in the solution. Cotton fabric padded with the solution, heated in a stream of air for 10 min. at 140°C., washed and air dried, was very strong and flame resistant.

USP 2,886,539

The polymers obtained by reacting a 1-aziridinyl phosphine oxide or sulphide with a methylol phosphorus compound of formula—

$$R{\cdot}CH_2{\cdot}PO{\cdot}(CH_2OH)_2 \text{ or } [(RCH)_2{\cdot}P{\cdot}(CH_2OH)_2]^+A^-$$

(R = OH or a radical formed by the reaction of the OH group of a PCH_2OH group with a N-compound containing at least one trivalent N atom and at least one H or CH_2OH attached to trivalent N; A = monovalent anion) are used in aqueous solution or dispersion as flame resistant agents. Thus cotton cloth was impregnated with an aqueous solution made up of tri(1-aziridinyl)phosphine sulphide (10%), tetrakis(hydroxymethyl)phosphonium chloride (10), triethanolamine (2) and methylol melamine (4). It was dried, baked for 3 min. at 160°C., washed and dried. The treated cloth was strong, had a good handle and was highly flame resistant. C.O.C.

Water Repellent Finishing Agent

Peter Spence & Sons *BP* 817,298

A water repellent composition for textiles comprises a hydrocarbon wax and the titanium ortho ester of iso-octanol. It is applied from white spirit solution and is particularly suitable for treating garments which contain absorbed detergent. C.O.C.

Silicon-Silica Emulsions for producing a Water-repellent and Soil-resistant Finish

DuP *USP* 2,881,146

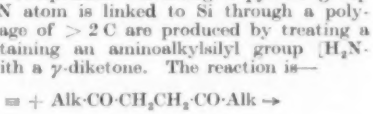
Organopolysiloxane emulsions containing a polymerisation catalyst and colloidal silica impart outstanding soil resistance without any lessening of the water-repellent finish given by the siloxane. C.O.C.

Pyrrole-containing Organosilicon Compounds

Union Carbide Corpn. *USP* 2,881,184

Organosilicon compounds containing a pyrrole group in which the N atom is linked to Si through a polymethylene linkage of > 2 C are produced by treating a compound containing an aminoalkylsilyl group [$H_2N{\cdot}(CH_2)_{>2}Si \equiv$ with a γ-diketone. The reaction is—

$$H_2N(CH_2)_{>2}\,Si \equiv + \text{Alk}{\cdot}CO{\cdot}CH_2CH_2{\cdot}CO{\cdot}\text{Alk} \rightarrow$$

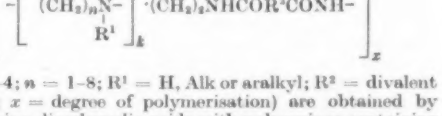


They are used as sizes, particularly for glass fibres, and as modifiers for thermosetting resins. C.O.C.

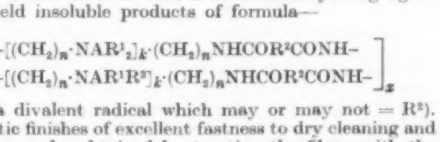
Water-soluble Basic Polyamides and their Alkylation Products as Antistatic Finishes

Onyx Oil & Chemical Co. *USP* 2,882,185

Soluble basic polyamides of formula—



(k = 1–4; n = 1–8; R^1 = H, Alk or aralkyl; R^2 = divalent radical; x = degree of polymerisation) are obtained by condensing dicarboxylic acids with polyamines containing two primary amino groups and one or more tertiary N atoms. When treated with a divalent alkylating agent they yield insoluble products of formula—



(R^3 = a divalent radical which may or may not = R^2). Antistatic finishes of excellent fastness to dry cleaning and washing can be obtained by treating the fibres with the water-soluble polyamide and then crosslinking them on the fibre by treatment with a divalent alkylating agent. C.O.C.

Water-repellent Agents

FBy *BP* 815,944

Aqueous emulsions containing paraffin wax, ester waxes and chromium complex salts of the Werner type containing acyl groups yield water-repellent finishes on textiles of very good fastness to washing with soap and soda if after washing the fabric is acidified with acetic acid before being dried. C.O.C.

Copper Antimonate as a Mildew-proofing and Flame-resisting Agent

Harshaw Chemical Co. *USP* 2,881,097

Copper antimonate is an effective mildew-proofing agent and when used in combination with halogenated organic compounds has flame resisting properties. C.O.C.

Synthetic Resin Binder Compositions for Pigments

Rohm & Haas Co. *USP* 2,883,304

Aqueous dispersions of linear copolymers of monoethylenically unsaturated molecules one of which is of formula $CH_2{:}CH{\cdot}O{\cdot}A{\cdot}NR^1CONHR^2$ (A = alkylene of 2–4 C or ether-oxygen linked alkylene of 2–3 C; R^1 and R^2 = same or different, H, aliphatic of 1–3 C or ether-linked aliphatic of 1–3 C or $R^1 + R^2$ together = $-CH_2-CH_2-$, $-CH(CH_3)CH_2-$ or $-(CH_2)_3-$) when pigmented and applied to textiles yield prints or dyeings of good fastness to washing. Thus the dispersion of the copolymer of *n*-butylacrylate and β-ureidoisobutyl vinyl ether (9:1) when pigmented with phthalocyanine blue (C.I. Pigment Blue 15) with addition of a little hydroxyethylcellulose, when padded into cotton cloth, dried at 240°F. and baked for 10 min. at 300°F. yields a dyeing which is fast to washing and to "cyclic ageing", i.e. exposure in a Fade-Ometer for 50 hr. and then subjecting to a washing test. C.O.C.

Pigment-binding Composition

Rohm & Haas Co. *USP* 2,886,474

Water-insoluble linear polymers containing $> 1\%$, preferably 5–25%, of a monomer of formula $CH_2{:}CR^1{\cdot}A{\cdot}$

(R^1 = H or CH_3; n = 0 or 1; A = $-\overset{O}{\overset{\|}{C}}-$ when n = 0 and either $-O-$, $-\overset{O}{\overset{\|}{C}}-O-$ or $-CO{\cdot}NR^5-$ (R^5 = H or satd. aliphatic hydrocarbon of 1–10 C) when n = 1; R^2 = alkylene of 2–10 C; R^3 and R^4 = same or different, H or satd. aliphatic hydrocarbon of 1–10 C), e.g. methacrylamide, when used in aqueous dispersion as binders for pigments for colouring textiles yield coloration of good fastness to washing. C.O.C.

Solutions of Nylon as Coating Compositions for Textiles, Paper, Metals, etc.

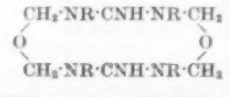
American Enka Corpn. *USP* 2,882,186

Nylon is readily soluble in an azeotropic mixture of 2-chloroethanol and water when it is heated to 80°C. the solution being thixotropic when cold. The mixture should be buffered to pH 6·8–9·2 to prevent formation of HCl. The solution can be used as a coating composition to yield tough, resilient smooth coatings. Addition of $> 3{\cdot}5\%$ trichloroethanol to the azeotropic mixture results in coatings of high gloss and unusual strength. C.O.C.

Adhesives for Binding Textiles to Rubber

United States Rubber Co. *BP* 815,860

The products formed by treating HCHO with compounds of formula $R^1NH{\cdot}CNH{\cdot}NHR^2$ (R^1 and R^2 = same or different Ar), e.g. 1,3-diphenylguanidine, have the formula—



(R = same or different Ar). Used in conjunction with resorcinol they are an excellent adhesive for bonding rubber to cellulosic or synthetic polymer textiles. C.O.C.

Ultraviolet Radiation Absorbents

Ciba *BP* 816,750

Colourless compounds containing one or more 1,3,4-oxdiazole or 1,3,4-thiadiazole rings and at least one

aromatic nucleus bound directly to one of these rings and which show little or no blue fluorescence, e.g. 2,5-bis-[2′-methoxyphenyl-1′)-]-1,3,4-oxdiazole, are used as ultra-violet radiation absorbers. C.O.C.

Polymethoxyaldehyde Compounds as Tanning Agents
U.S. Secretary of Agriculture *USP* 2,880,053

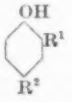
Polymethoxyaldehydes of formula $CH_3{\cdot}(CH(OCH_3){\cdot}CH_2)_{2-20}{\cdot}CHO$, obtained by hydrolysis of the corresponding acetals obtained, e.g. by condensing methyl vinyl ether and methanol, are good substitutes for vegetable tanning agents. C.O.C.

Mordants for Acid Dyes used in Photography
Eastman Kodak Co. *USP* 2,882,156

Strongly basic products readily soluble in dilute acids are obtained by condensing a polyvinyl-oxo-compound, e.g. poly(vinyl methyl ketone), with aminoguanidine. They are compatible with hydrophilic colloidal materials, e.g. gelatin, and readily form water-insoluble salts with acid dyes. They are very useful in preventing acid dyes from diffusing into other layers and in imbibition printing blanks, to prevent matrix poisoning, i.e. transfer of some undesirable agent from the blank to the matrix. C.O.C.

Solvents for Colour Couplers
Kodak *BP* 820,329

Compounds of formula—



(either R^1 or R^2 = $-(CH_2)_mCH_3$ and the other = $-(CH_2)_n{\cdot}CH_3$ or $-CO(CH_2)_nCH_3$; n and m = same or different, 0–17, but $n + m$ = 7–17), e.g. 2,4-di-*n*-amylphenol and 2-*n*-valeryl-4-*n*-amylphenol, when used as solvents for colour couplers cause no fading of the dye image and do not become discoloured. They diffuse less and are more stable to oxidation than those previously used. C.O.C.

Poly(Vinyl Chloride) Copolymer/Polyurethane Primers for Vinyl Resin Coatings
Union Carbide Corpn. *BP* 820,145

A resinous primer for vinylchloride resin coatings on nylon, glass fibres, metal, etc., comprises a polyurethane resin and a copolymer of vinyl chloride, a vinyl carboxylate and maleic acid or anhydride. C.O.C.

2-Vinylpyridine— Versatile Intermediate (IV this page)
Differences and Analogies in Chemical Structure of Syntans and Leather Dyes (IV p. 553)
Constitution of the Colouring Matter of Spruce Bark, a New Class of Condensable Tannin (IV p. 558)
Organosilicon Compounds in Cotton Finishing (X p. 573)

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

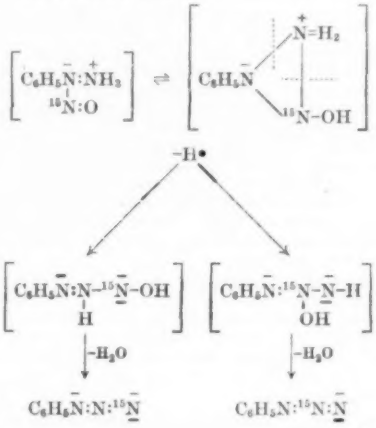
Steric Effects in the Catalytic Reduction of Nitro Compounds of an Aromatic Series
F. N. Mazitova and Ya. M. Paushkin
Doklady Akad. Nauk S.S.S.R., **125**, 1033–1036 (11 April 1959)

Seven different nitroalkylphenols are reduced with hydrogen (Pt catalyst) in alcohol at 20°c. It is found that the rate of reduction decreases with an increase in the size and complexity of the alkyl substituent, and an explanation based on steric effects is given. G.J.K.

Life and Times of Peter Griess
W. H. Cliffe J.S.D.C., **75**, 278–285 (June 1959)

Reactions with ^{15}N. XXXII— Formation and Decomposition of Nitrosophenylhydrazine
K. Clusius and K. Schwarzenbach
Helv. chim. Acta, **42**, 739–748 (4 May 1959)

The diazotisation of phenylhydrazine with labelled nitrous acid, $H^{15}NO_2$, yields a mixture of the isotopic isomeric azides $C_6H_5N{:}^{15}N{:}N$ and $C_6H_5N{:}N{:}^{15}N$. The intermediate cpd. in this reaction is now proved to be the labelled α-nitrosophenylhydrazine (I), $C_6H_5\overset{\alpha}{N}(^{15}NO){:}\overset{\beta}{N}H_2$, and not its β-isomeride, $C_6H_5\overset{\alpha}{N}H{:}\overset{\beta}{N}H(^{15}NO)$. Preparatively, only I is isolable, and it undergoes thermal decomp. in bromobenzene to aniline and labelled nitrous oxide, $N^{15}NO$. Despite its unique isotopic constitution, I decomp. in aq. acid soln. to give both the isomeric azides above, a reaction interpreted as a removal of H_2O from a tautomeric kation of I according to the scheme below. Similar results from the diazotisation of glycocoll and the transformation of *N*-substituted nitrosobenzylhydrazines support the proposed scheme—



H.H.H.

2-Vinylpyridine— Versatile Intermediate
E. R. Wallsgrove
Mfg. Chemist, **30**, 206–8, 210 (May 1959)

An account of the physical and chemical properties of 2-vinylpyridine now being produced in the U.K. on a commercial scale. It has a wide range of uses including that of an intermediate for dyes and wetting agents. 67 references. C.O.C.

Relative Rates of Reduction of a Series of Azo Compounds
G. P. Warwick J.S.D.C., **75**, 291–299 (June 1959)

Mechanism of the Coupling Process
W. Bradley J.S.D.C., **75**, 289–291 (June 1959)

Recent Work on the Mechanism of Diazotisation
J. H. Ridd J.S.D.C., **75**, 285–289 (June 1959)

Excitation Energy Transfer from Dye Molecules in the Metastable State
R. Baver, A. Baczyński, and M. Czajkowski
Bull. Acad. polon. sci., Ser. sci. Math., astron. et phys., **6**, 653–658 (1958): *Chem. Abs.*, **53**, 5867 (10 April 1959)

A paper, in English, giving the degrees of polarisation of fluorescence and phosphorescence respectively for Acridine Yellow in H_3BO_3 at concentrations from 5×10^{-6} to 1×10^{-2} g./l. and compared with the Jabłoński theory (*Chem. Abs.*, **52**, 7854 (1958)). The results are given in terms of the emission anisotropy, r, as defined by Jabłoński. At very low concentrations r is the same for both fluorescence and phosphorescence. Energy levels are considered and it is concluded that from the metastable level M transitions take place directly and not through an intermediate stage. C.O.C.

Metastable State of Dye Molecules
A. Jabłoński *Bull. Acad. polon. sci., Ser. sci. Math., astron. et phys.*, **6**, 589–593 (1958): *Chem. Abs.*, **53**, 5866 (10 April 1959)

An article, in English, in which transitions with two quantum numbers, p and n, are considered for luminescent molecules with $2p\pi$ "metallic" electrons. The metastable energy level, M, is believed to be the lowest one of the set p_3, n = 1,2,3 . . . . The transition $M \rightarrow N$ (N being the ground level, p_1, n = 1) is forbidden. Low or negative polarisation of the fluorescence M–N band is explained. Probably $p = p_1$ corresponds to $2p\pi$ and $p = p_3$ to $3s\sigma$ electrons. The cases of Fluorescein and Trypaflavine are discussed. C.O.C.

Classifying Organic Dyes by Photoelectric Behaviour
H. Meier *Z. wiss. Phot.*, **53**, 1–18 (1958):
Chem. Abs., **53**, 6783 (25 April 1959)

Organic dyes can have one of two types of photoconductive behaviour, the two differing in the effect of O in absence of moisture. Vacuum-photoconductors are photoconductive *in vacuo*, the effect diminishing in an atmosphere of O, whereas O-photoconductors show increase in photoconductivity with increase in the pressure of O, their photoconductivity in some cases becoming immeasurably small in absence of O. Dyes of the fluorescein class, azo dyes, acridine dyes, some cyanine dyes, etc. are vacuum-photoconductors, while those of the malachite green and fuchsine, rhodamine, thiazine, oxazine classes, and some cyanine dyes are O-photoconductors. Analogy with inorganic photoconductors suggests that the organic vacuum-photoconductors show electron photoconductivity and the O-photoconductors positron hole photoconductivity; in some dyes, e.g. Malachite Green (C.I. 42000), both types of carriers seem to be formed on exposure to light. The difference in the nature of the conduction process in dyes may be connected with the type of intermolecular association within the crystal. The measurements were made with cells having reticule electrodes made by "electric etching" of Rh on glass.
C.O.C.

Construction and Transformations of the Diazo Linkage
B. V. Passet and B. A. Poraĭ-Koshits
Trudy Leningrad. Tekhnol. Inst. im. Lensoveta, **46**, 133–146 (1958):
Chem. Abs., **53**, 12153 (10 July 1959)

Potentiometric titrations confirm that the diazo cation is an aprotic, dibasic acid, while the diazo anion is a diprotic base. Neutralisations of these ions are both two-step processes. That of the cation involves a slow first-step whose product, diazo hydrate, reacts almost instantly with a second equivalent of alkali to give the diazo anion. In the anionic neutralisation, the first step is quicker and leads to an intermediate in which the diazo linkage forms tautomeric equilibrium between the diazo hydrate and nitrosamine. Although the tautomeric equilibrium lies on the side of nitrosamine, the weaker acid, the mixture gradually passes over into the diazo cation. Thus the anion and cation are reversibly convertible through hydrolysis and the intermediate tautomeric mixture may be neglected in most cases. The two ions seem to have similar bonding systems and their incorporation into molecules seems to occur with a transfer of the reaction centre; however, their respective reactions with acid or alkali are surmised to be subject to catalysis and to involve direct transfer of the reaction centre.
C.O.C.

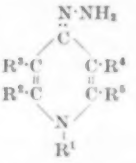
Substantivity of Acylacetanilides for Cellulose. I and II
G. Cordella *Boll. sci. fac. chim. ind. Bologna*, **16**, 10–13, 14–17 (1958):
Chem. Abs., **53**, 9679 (25 May 1959)

The affinity for cotton of various derivatives of acetanilide in 10, 20, and 30% solutions of NaCl decreases in the following order: 2-benzoyl-*p*-nitro- > 2-thenoyl-*p*-chloro- > 2-benzoyl-*p*-chloro- > 2-benzoyl-*m*-nitro- > 2-furoyl-*p*-chloro- > 2-benzoyl-*m*-chloro- > 2-benzoyl-*o*-chloro- > 2-thenoyl- > 2-benzoyl-*p*-methyl- > 2-benzoyl-*m*-methyl- > 2-benzoyl-*o*-methyl- > 2-furoyl- > 2-benzoyl-. The 2-acetyl- and 2-acetyl-*p*-chloro-derivatives and *N*-benzyl-2-benzoylacetamide have no affinity for cotton.
C.O.C.

Azo Dyes Formed by Oxidative Coupling
S. Hünig and G. Köbrich

V—Synthesis of 1-Substituted 4-Pyridonehydrazones
Ann., **617**, 181–202 (1958):
Chem. Abs., **53**, 7168–7169 (25 April 1959)

Various 1-substituted-4-pyridones were converted into quaternary salts and reacted with benzoylhydrazine to give benzoylhydrazones, which were hydrolysed with boiling aq. HCl to give the hydrazones—



(R^2, R^3, R^4, R^5 = H, Alk, Ar or NO_2; R^1 = Alk or Ar). With *p*-nitrobenzaldehyde in alcoholic ammonia these condense to give the *p*-nitrobenzylideneazines.

VI—Coupling 1-Substituted-4-pyridonehydrazones with Phenols
Ibid., 203–210:
Chem. Abs., **53**, 7170–7171

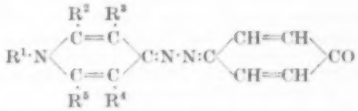
1-Substituted-4-pyridonehydrazones when oxidised with $K_3Fe(CN)_6$ in presence of phenol gave low yields, whereas α-naphthol and its substituents gave good yields of azo compounds.

VII—Coupling 1-Substituted-4-pyridonehydrazones with Aromatic Amines
Ibid., 216–220:
Chem. Abs., **53**, 7171

Oxidative coupling of 1-substituted-4-pyridonehydrazones with amines such as *NN*-dimethylaniline and α-naphthylamine gave azo compounds, but reaction conditions were critical, and yields often low. E.S.

Colour and Constitution. VI—Pyridone azinobenzoquinones
S. Hünig and G. Kobrich *Ibid.*, 210–216:
Chem. Abs., **53**, 7169–7170

The 1-subst. 4-pyridonehydrazones of the previous abstract with $NaClO_4$ and benzoquinone gave the perchlorates of azo dyes, which with formic acid followed by addition of conc. aq. NaOH gave pyridoneazinobenzoquinones—



The colours of these compounds are discussed. E.S.

Effects of the Alkyl Groups in Fast Blue RR-type Azoic Diazo Components. I and II
J. Nishino, A. Nishikura, N. Kuroki, and K. Konishi
Yūki Gōsei Kagaku Kyōkaishi, **17**, 159–166 (1959):
Chem. Abs., **53**, 8631–8632 (10 May 1959)

Homologues of C.I. Azoic Diazo Component 24 (2-benzamido-5-aminohydroquinone dimethyl ether) and of Violet Base Ciba III in which the methyl groups were replaced by ethyl, propyl, and butyl groups were prepared and compared in dyeing and fastness properties. No advantages over existing commercial products were apparent. E.S.

Method of Preparation of Lake Red S and Related Pigments
E. A. Titov, V. F. Borodkin, and R. A. Maksimova
Trudy Ivanovsk. Khim. Tekhnol. Inst. **1956**, No. 5, 190–193:
Referat. Zhur. Khim. **1957**, Abstr. No. 13188:
Chem. Abs., **53**, 11842 (25 June 1959)

Ca lakes such as C.I. Pigment Red 49 were prepared by carrying out diazotisation, coupling and laking simultaneously, but consumption of reagents was higher than in a conventional synthesis. E.S.

Azo Dyes
K. Murata and K. Harada

XIII—Azo Dyes from 6-chloro-2,4-dimethoxyaniline and 4-Amino-2-chlorophenetole
Hiroshima Daigaku Kōgakabū Kenkyū Hōkoku, **7**, 101–110 (1958):
Chem. Abs., **53**, 11839 (25 June 1959)

HCl passed through a cooled mixture of *p*-nitrophenol, methanol, and diethyl ether give 6-chloro-2,4-dimethoxyaniline which was diazotised and coupled with naphthol- and naphthylamine-sulphonic acids to give monoazo acid dyes; e.g. Schaeffer acid gave a red.

Treatment of *p*-nitrophenetole in a mixture of acetic and hydrochloric acids with $KClO_3$, followed by reduction, gave 4-amino-2-chlorophenetole, which was similarly diazotised and coupled to give similar dyes.

XIV— Azo Dyes from 2-Amino-5-anilinophenylthioglycollic Acid *Ibid.*, 111–113: *Chem. Abs.*, **53**, 11841

Sulphur chloride reacted with aniline hydrochloride at 50°c. to give 5-chloro-2,1-phenylenethiazthionium chloride which with aniline gave the 5-anilino compound. Chloracetic acid then gave 2-amino-5-anilinophenylthioglycollic anhydride, which was diazotised and coupled with naphtholsulphonic acids to give 4-anilino-2-carboxymethylthiophenylazo compounds.

XV— Azo Dyes from Benzidine-3,3′-dicarboxylic acid *Ibid.*, 115–117: *Chem. Abs.*, **53**, 11841

Reduction of *o*-nitrobenzoic acid with Zn in aq. NaOH gave benzidine-3,3′-dicarboxylic acid, which was tetrazotised and coupled with naphtholsulphonic acids to give disazo dyes, e.g. G acid gave a compound dyeing silk pink to violet.

XVI— Azo Dyes from 2-Nitrobenzidine *Ibid.*, 119–125: *Chem. Abs.*, **53**, 11841

Nitration of benzidine gave the 2-nitro compound which was tetrazotised and coupled with, e.g. H acid, which gave a dark red disazo dye for wool. E.S.

Azoic Dyes containing the Fluorene Ring
N. Ishikawa, N. Izawa, and M. Okazaki
J. Soc. Textile Cellulose Ind., Japan, **15**, 209–213 (March 1959)

Couplers containing the fluorene ring were examined for their use in cotton dyeing. One series used contained 3-hydroxyfluorene-2-carboxylic acid, anilide, *o*- or *m*-toluidide or *o*- or *p*-anisidide. They developed fully only when the pH of the bath was near 7. They yielded good olives of good fastness to washing and satisfactory fastness to light. The other series of couplers contained fluoreneamides of 2-hydroxy-3-naphthoic acid. The 3-fluoreneamide and 3-methoxy-2-fluoreneamide gave much better dyeings than the 2-fluoreneamide or 7-methoxy-2-fluoreneamide. The dyeings had good fastness to light and excellent fastness to washing. C.O.C.

Dyeing Properties of Acid Azo Dyes— II
M. Sekido and T. Iijima
J. Soc. Textile Cellulose Ind., Japan, **15**, 214–216 (March 1959)

The dyeing properties of dyes of the type obtained by alkaline (in 2-position) and acid (in 8-position) coupling of diazotised *p*-substituted aniline and γ-acid were investigated where the *p*-substituent was H, SO_3H, SO_2NH_2, COOH, $COOC_2H_5$, Cl, CH_3, OCH_3, OC_2H_5, NO_2, NH_4, $NHCOCH_3$ and NHC_6H_5. The absorption curves of aqueous solutions of dyes obtained by alkaline coupling (I) and those obtained by acid coupling (II) are very differently shaped. I have 2 absorption maxima and II have only 1 maximum. Dyeings of I on wool are much duller than those of II. The rate of dyeing of II on to wool from a boiling bath is much quicker than that of I. All the dyes had considerable affinity for wool from a neutral bath. Dyeings of II had better light fastness than those of I, the contrary being true for fastness to washing. C.O.C.

Preparation and Oxidation-reduction Potentials of Variamine Blue Derivatives
L. Erdey, É. Bányai, E. Zalay, and M. Téry
Acta Chim. Acad. Sci. Hung., **15**, 65–79 (1958): *Chem. Abs.*, **53**, 6119 (10 April 1959)

Substitution of various groups in the 4-amino-4′-methoxydiphenylamine or Variamine Blue molecule results in change in the standard oxidation potential which is in accord with the known electron attracting or repelling properties of these groups. C.O.C.

Metal Derivatives of Aryl Azo Pyrazolone Dyes. III— Molarity Quotients of *p*- and *m*-Substituted Pyrazolone Dyes
F. A. Snavely and B. D. Krecker
J. Amer. Chem. Soc., **81**, 4199 and 4200 (20 Aug. 1959)

Potentiometric measurement of the relative stabilities of simple aryl azo pyrazolone dyes in 75 vol. % dioxane confined the order of decreasing stability Cu > Ni > Co > Zn obtained in previous work. Stability of the Cu derivatives of the *p*-substituted dyes increases in the order $NO_2 < (Cl, Br) < I < OCH_3 < CH_3 < H$. C.O.C.

Affinities, Heats of Dyeing and Entropy Changes of Some Blue Direct Dyes
T. Urahata
Kôgyô Kagaku Zasshi, **60**, 1468–1475 (1957): *Chem. Abs.*, **53**, 14522 (10 July 1959)

The following dyes were prepared by column chromatography using pure cellulose and aqueous pyridine as developer: Diamine Black RO (C.I. Direct Black 29), Azo Blue (C.I. Direct Violet 28), Benzo Azurine G (C.I. Direct Blue 8), Chrysamine G (C.I. Direct Yellow 1), Sirius Violet BB (C.I. Direct Violet 51), Brilliant Blue 5R, Oxamine Sky Blue 5B, Oxamine Sky Blue 6B, Direct Blue 2B (C.I. Direct Blue 6), Benzo New Blue 5B (C.I. Direct Blue 25), Direct Brilliant Blue G, Sirius Supra Blue BRR (C.I. Direct Blue 71), Sirius Supra Blue G (C.I. Direct Blue 78), and Diamine Fast Yellow A (C.I. Direct Yellow 11). Their affinity, heat of dyeing and entropy charge were calculated by use of Vickerstaff's formula. The relationship of dye structure and their degree of basicity to affinity for cotton is discussed. C.O.C.

Metal Derivatives of Azo Pyrazolone Dyes. II— Molarity Quotients of Azo Pyrazolone Dyes containing *o*-Methoxy and *o*-Thiomethoxy groups
F. A. Snavely, B. D. Krecker, and C. G. Clark
J. Amer. Chem. Soc., **81**, 2337–2342 (20 May 1959)

Potentiometric measurement in 75 vol. % dioxane of the relative stabilities of the metal derivatives of the title dyes shows that the order of decreasing stability of the metal derivatives, Cu > Ni > Co > Zn > Cd agrees with the order already recorded by Snavely *et al.* (J.S.D.C., **73**, 492 (1957)) for other terdentate dye groups. The S of the thiomethoxy group is a much stronger donor atom than the O of the methoxy group. The log Q_1 values are generally greater than the log Q_2 values, the converse to that for the simple bidentate azo pyrazolone dyes. C.O.C.

Metal Derivatives of *o*-Hydroxy Azo Compounds III— Preparation, Properties and Relative Stability Constants of Cobaltous Chelates of Phenylazo-*p*-cresols
Y. Hayakawa and H. Kido
Sci. Repts. Saitama Univ., **3A**, 33–39 (1958): *Chem. Abs.*, **53**, 11072 (25 June 1959)

Preparation, composition and qualitative solubilities are described of 9 Co(II) chelates of phenylazo-*p*-cresols. They were prepared by dissolving the azo dye in hot absolute alcohol and then adding 0·1 mole/l. of $Co(OOCH_3)_2$ solution the pH being controlled by NH_4OH. The mixture was refluxed in a N atmosphere and the precipitate filtered off. In this way the following were prepared: Co(II) bis[2-(3-tolylazo)-4-methylphenolate], Co(II) bis[2-(4-tolylazo)-4-methylphenolate], Co(II) bis[2-(4-chlorophenylazo)-4-methylphenolate], Co(II) bis[2-(2-nitrophenylazo)-4-methylphenolate] and Co(II) bis[2-(4-bromophenylazo)-4-methylphenolate]. These complexes seem to be more soluble than the corresponding Cu and Ni complexes, while their stability constants almost equal those of the Ni complexes but are less than those of the Cu complexes. 17 references.

IV— Absorption Spectra in the Near Ultraviolet and Visible Region of Copper, Nickel and Cobaltous Chelates of Phenylazo-*p*-cresols
Y. Hayakama and K. Hiriguchi *Ibid.*, 417: *Chem. Abs.*, **53**, 11072–3 (25 June 1959)

Tables give the absorption spectra at 230–600 mμ. of 17 phenylazo-*p*-cresol dyes and of their Cu, Ni and Co complexes. In the first absorption band the wavelength of azobenzene (430 mμ.) is longer than that of the *o*-hydroxyphenylazo-*p*-cresols (385–400 mμ.) but in the 2nd band they resemble each other. The difference between wavelengths in the 1st band is caused by the effect of H or co-ordination bonds in the *p*-cresols and the metal complexes on the N:N group. Maximum absorptions of the Cu and Ni complexes of the phenylazo-*p*-cresols are near 380, 330 and 250 mμ. Those of the Co complexes are somewhat different being about 400 mμ. in the first band, somewhat similar in the second band, about 10% longer in the third. C.O.C.

Red Organic Pigments

III— Influence of Moisture Content on the Colour of the Pigments

N. Nakajima and K. Tsumeji

Shikizai Kyokaishi, **30**, 2–6 (1957)

The red pigment $[CH_3(Cl)(SO_3^-)C_6H_2N{:}NC_{10}H_5(OH)COO^-]Ca^{++}$ was dried and then moistened at 25°c. Another sample was dried at 75°c. and moistened at 25°c. When dried, the colour deepened and changed reversibly at a moisture content of 1·5–4·0% in both cases. No difference in crystal shape was seen between the moist and dry pigment and so the colour change seems to be caused by the water absorbed.

IV— Electrophoresis

N. Nakajima and H. Hirohata *Ibid.*, 52–54:

Chem. Abs., **53**, 4768 (10 March 1959)

Electrophoretic mobilities were determined with a U-tube electrophoretic cell. Pigment, % extender ($BaSO_4$), % metallic soap, electrophoretic mobility (cm./v.sec.) and ζ potential (mv.) were: Permanent Red 4R (yellowish), —, —, 2·38, — 38; Permanent Red 4R (red), —, —, — 2·42, — 40; Permanent Red 10B (Ba salt), 40, Turkey red oil soap 5, — 2·90, — 31; Permanent Red 10B (Ca salt) 40, Turkey red oil soap 5, — 3·52, — 36; Permanent Red F5R (C.I. Pigment Red 48) A, 25, rosin soap 5, — 3·06, — 32; Permanent Red F5R B, 25, rosin soap 10, — 2·70, — 30; BON Maroon Light (C.I. Pigment Red 58), —, —, — 4·00, — 45; BON Maroon Red Light (C.I. Pigment Red 48), —, —, — 3·87, — 40; Lithol Red B, 40, 5, — 3·45, — 38. The experimental conditions for the first three pigments were pH 7·0, 20°c. and 3% pigment and for the remainder pH 6·0, 30°c. oil 2% pigment. The electric charge of Permanent Red F5R A was positive in presence of a cationic surfactant but negative in presence of other types of surfactants. C.O.C.

Absorption Spectra of Hydrophobic Dyes and Use of these Dyes in Light Filters

E. A. Veller and B. A. Poraĭ-Koshits

Trudy Leningrad. Tekhnol. Inst. im. Lensoveta, **30**, 3–33 (1954):

Chem. Abs., **53**, 11985 (10 July 1959)

Spectrophotometric characteristics of 84 hydrophobic dyes were determined in the ultraviolet, infrared and visible ranges of the spectrum. Hydrophobic disperse and solvent dyes that are soluble in non-polar solvents obey Beer's law when their initial concentration charged in ratio 1 : 500 or 1 : 1000. All organic solvents with rare exceptions have little effect on the damping coefficient of hydrophobic dyes in the visible and ultraviolet spectrum regions. The solubility of hydrophobic dyes in polar solvents is low (0·001% in alcohol), in non-polar solvents in most cases it is 1–3% and in some cases 12–15%. Maximum solubility was observed in dichloroethane. The data given enable ready calculation of the concentration of dyes for light filters. C.O.C.

Coloristic Evaluation of Reactive Dyes

M. Kalous *Textil.*, **14**, 28–30 (1959):

Chem. Abs., **53**, 14521 (10 July 1959)

Laboratory trials indicate that Cibacron (Ciba) and Procion H (ICI) dyes are, with one exception, identical. C.O.C.

Differences and Analogies in Chemical Structure of Syntans and Leather Dyes

E. Krzywicki *Przegląd Skórzany*, **12**, 291–197 (1957):

Chem. Abs., **53**, 14562 (10 July 1959)

Review, 15 references. C.O.C.

Absorption Spectra of Methylene Blue (C.I. 52015) and Toluidine Blue (C.I. 52040)

B. P. Nikol'skiĭ and V. V. Pal'chevskiĭ

Zhur. Fiz. Khim., **32**, 2123–2128 (1958):

Chem. Abs., **53**, 6753 (25 April 1959)

Addition of neutral salts (KCl) caused shift in the absorption maximum of Methylene Blue and in optical d. *D* of Methylene Blue was linearly related to the ionic strength of the solution. The molecular coefficient of *D* at zero ionic strength is suggested as a characteristic value of the dye. The situation of the maximum in the ultraviolet range was unaffected by the ionic strength of the solution. The Toluidine Blue cation absorption spectrum was similar to that of Methylene Blue but its absorption maxima were at shorter wavelengths and presence of isomers caused the maxima to be less definite: because of this the effects of the ionic strength on solutions of Toluidine Blue could only be studied qualitatively. C.O.C.

Oxidation Mechanism of Malachite Green (C.I. 42000)

V. Hanousek and M. Matrka

Chem. Listy, **52**, 1774–1779 (1958):

Chem. Abs., **53**, 6146 (10 April 159)

The oxidative fission of C.I. 42000 to BzOH and diphenoquinone 4,4′-bisdimethylammonium derivative is intramolecular and proceeds by way of a labile cyclopropane derivative. Evidence supporting this mechanism is given. C.O.C.

Preparation and Study of Leucocyanides of Triarylmethane Dyes

D. Tobin, A. Katzakian, and M. Chisholm

U.S. Army Med. Research Lab., Rept. No. 292, 10 pp. (1957):

Chem. Abs., **53**, 12232 (10 July 1959)

Leucocyanides were prepared from 5 g. basic triarylmethane dye and 1–2 g. NaCN in 150 ml. hot $(CH_3)_2SO$. The resultant solution was stirred, filtered, cooled, poured into ice and water, the crude leucocyanide filtered off, dissolved in 100 ml. 6 N-HCl, diluted with 200 ml. water, the solution treated repeatedly with 3 g. portions of carbon until the filtrate was nearly colourless, the filtrate diluted to 500 ml., cooled, made alkaline with conc. NH_4OH, filtered, the precipitate washed with hot water, dissolved in 100 ml. hot $(CH_3)_2SO$, 0·2 g. NaCN added, the solution filtered, cooled, poured into ice and water, filtered, the precipitate washed with hot water, and dried in a vacuum desiccator over $CaCl_2$. If the product was not white, it was dissolved in 100 ml. benzene, treated with carbon and precipitated by adding petroleum ether. Leucocyanides were prepared from the following dyes (m.p. and % yield given): Malachite Green (C.I. Basic Green 4) 178–9, 92·4; Rhoduline Blue 6GA (C.I. Basic Blue 1) 197–8, 31·2; Brilliant Green (C.I. Basic Green 1) 159–160, 75·1; Pararosaniline (C.I. Basic Red 9) 242–4, 82·2; Magenta XX (C.I. Basic Violet 14) 249–250, 70·2; Magenta ABN (C.I. Basic Violet 2) 249–250, 58·5; Dahlia 181–2, 81·2; Methyl Violet (C.I. Basic Violet 1) 254–5, 73·5; Crystal Violet (C.I. Basic Violet 3) 289–290, 71·7; Ethyl Violet (C.I. Basic Violet 4) 224–5, 79·2; Spirit Blue (C.I. Solvent Blue 3) 185–6, 31·2; Victoria Blue 4R (C.I. Basic Blue 8) 159–161, 41·4; Victoria Blue R (C.I. Basic Blue 11) 250–1, 85·4; Night Blue (C.I. Basic Blue 15) 239–240, 50·7. Evidence was obtained supporting the mechanism proposed by Harris *et al.* (*Chem. Abs.*, **29**, 6143 (1935)) for the formation of a coloured ion followed by fading where an alcoholic solution of Malachite Green leucocyanide is subjected to ultraviolet radiation. A mechanism is proposed for the similar behaviour of Victoria Blue R leucocyanide. C.O.C.

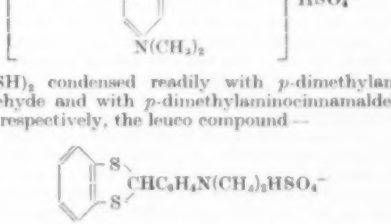
Dyes having a Sulphenium Structure

O. Neunhoeffer and A. Nowack

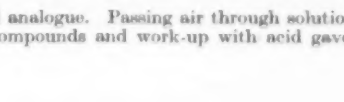
Naturwissenschaften, **45**, 491–492 (1958):

Chem. Abs., **53**, 12681 (10 July 1959)

2 mol. methylthiophenylmagnesium bromide reacted with 1 mol. *p*-dimethylaminobenzoic acid ester gave, after work-up in presence of dil. H_2SO_4, the acid sulphate of a Crystal Violet analogue—



o-$C_6H_4(SH)_2$ condensed readily with *p*-dimethylaminobenzaldehyde and with *p*-dimethylaminocinnamaldehyde to give, respectively, the leuco compound—



and its styryl analogue. Passing air through solutions of these leuco compounds and work-up with acid gave the

corresponding dyes. o-$C_6H_4(SH)_2$ with chloromalonaldehyde gave bis[1,3-benzo-dithio-2-yl]-chloromethane which in wet $CHCl_3$ solution gave a red salt when air was passed through. Bromomalonaldehyde behaved similarly. C.O.C.

Synthesis of Quinonaphthalonesulphonic Acid
Y. Yamakazi, T. Ishii, and K. Shiro
Yûki Gôsei Kagaku Kyôkai Shi, **17**, 229–231 (1959): *Chem. Abs.*, **53**, 14105 (10 July 1959)

Naphthalic anhydride (24 g.) was heated 8 hr. with quinarsine (20) and a little Zn dust at 190–5°c., washed with dil. HCl and then with hot 5% aq. Na_2CO_3 to give the yellow quinonaphthalone (I) m.p. 261·2–262·0°c. I (13·6 g.) in 25% H_2SO_4 (210) was heated 90 min. at 68–70°c., cooled, poured into ice water, filtered and the filtrate neutralised with NaOH to give Na quinonaphthalone sulphonate (II). Quinarsine (3 g.) and $C_6H_4(CO)_2$ (45) were heated together for 4 hr. at 180–190°c., cooled, powdered, washed with 60 c.c. warm 10% HCl and then with 100 c.c. 10% Na_2CO_3 to give the yellow quinophthalone (5·3), m.p. 237·0–237·5°c. This (5) in 25% fuming H_2SO_4 (10) was heated 2 hr. at 48–50°c. and 5 hr. at 60–5°c., cooled, poured into ice water and neutralised with 10% NaOH to give Quinoline Yellow (C.I. 47005) (8·8). The dyeing properties and fastnesses of Quinoline Yellow and Na quinonaphthalonesulphonate are compared. C.O.C.

Rydberg Series in Dyes. III— Measurement of Anisotropy and Classification of Electronic Bands in the Simplest Cyanines
G. Scheibe, J. Kern, and F. Dörr
Z. Elektrochem., **63**, 117–121 (1959): *Chem. Abs.*, **53**, 6753 (25 April 1959)

Study of the ultraviolet absorption spectrum of 1,5-dipyrrolidylpentamethin perchlorate has enabled demonstration of 4 electronic transitions and the probable existence of a fifth one. Depending on polarisation they are classified as $A_1 \rightarrow A_2$ and $A_1 \rightarrow B_1$ transitions. The variation of the relative intensities of the vibrational structure of the longest wavelength transition in a homologous series of dyes suggests that the π-bond system is loosened in the excited state, the more so the shorter the methin chain. C.O.C.

Coal-tar Colours. XXII— 4,5-Dibromofluorescein (C.I. 45370A) and Related Bromofluoresceins
C. Graichen and J. C. Molitor
J. Assoc. Offic. Agr. Chemists, **42**, 149–160 (1959): *Chem. Abs.*, **53**, 9573 (25 May 1959)

Paper and column chromatography and spectrophotometry show that direct bromination of fluorescein yields 4,5-dibromofluorescein, 4-bromo-(I), 2,4-dibromo-(II), 2,5-dibromo- (III), 2,4,5-tribromo- (IV) and 2,4,5,7-tetrabromofluoresceins (V). Other syntheses were used for higher yields of II, III and for 2-bromo- (VI), 2,7-dibromo- (VII), and 2,4,7-tribromofluoresceins (VIII). Commercial samples (20) of 4,5-dibromofluorescein (D&C Orange No. 5) contained I, II, III, IV, and V as impurities but no VI, VII, or VIII. C.O.C.

Fluorescent Derivatives of 1,2,3-Triazole. VII— Sodium Salts of Some Di- and Tritriazolesulphonic Acids
J. Dobáš, J. Pirkl and, V. Hanousek
Chem. Listy, **52**, 1310 (1958): *Chem. Abs.*, **53**, 9195 (25 May 1959)

Na 2-(4′-sulphophenyl)-5-aminobenzotriazole diazotised and coupled with $H_2NC_{10}H_6SO_3Na$ followed by dissolving the resulting dye in an aqueous solution of ammonia and pyridine and oxidising with ammoniacal $CuSO_4$ yielded di-Na 2-(2′-phenyl-5′-benzotriazolyl)naphtho[1,2]triazole-4″,6-disulphonate. Similarly prepared were di-Na 2-(2′-phenyl-5′-benzotriazolyl)naphtho[1,2]triazole-6,8-disulphonate, di-Na 2-[2′-(1″-naphthyl)-5′-benzotriazolyl]-naphtho[1,2]-triazolyl-4″,6-disulphonate, tri-Na 2-(2″-phenyl-5″-benzotriazolyl)-4-methyl-5-[2′-naphtho[1,2]-triazolyl]benzotriazole-4″,6′,7-trisulphonate and di-Na 2-(2′-phenyl-5′-benzotriazolyl)-5-phenyl-ang-benzobis-triazole-4″,4‴-disulphonate. These compounds are faster to light on cotton than are bistriazoles prepared from *p*-phenylenediamine and benzidine and derivatives but less fast than benzo- and naphthotriazoles based on stilbene. C.O.C.

Influence of Structure and of Some Physicochemical Factors on the Sensitising Effect of Polymethin Dyes
I. I. Levkoev, E. B. Lifshits, S. V. Natanson, N. N. Sveshnikov and Z. P. Sytnik
Wiss. Phot., Intern. Konf. Köln, 1–4 (1956): *Chem. Abs.*, **53**, 6843 (25 April 1959)

An investigation of the influence of the structure of a series of cyanine, merocyanine, and rhodacyanine dyes on their sensitising effect. Absorption spectra of the dyes at several concentrations are given and the influence of the structure on sensitisation is indicated in tables. C.O.C.

Polyene Aldehydes. I— Polymethin Dyes
M. Richter and P. Boyde
J. prakt. Chem., **7**, 184–190 (1950): *Chem. Abs.*, **53**, 10766 (10 June 1959)

A series of dyes containing an odd number of methin groups but no quaternary N atom and of formula $R^1(CH{:}CH)_n{\cdot}CH{:}CH{\cdot}S{\cdot}C(:S){\cdot}NR^2{\cdot}CO$ (n = 5–11; R^1 = CH_3, C_6H_5, furyl or naphthyl; R^2 = H or C_2H_5) was prepared by condensing various polyene aldehydes with rhodanine or *N*-ethylrhodanine in methanol or ethanol with piperidine as catalyst. C.O.C.

Interdependence of Colour and Structure of Some Dyes formed in Colour Development
I. I. Levkoev, B. S. Portnaya, I. A. Solov'eva, N. S. Spasokukotskii, and N. F. Turitsyna
Wiss. Phot., Intern. Konf. Köln, 477–484 (1956): *Chem. Abs.*, **53**, 6847 (25 April 1959)

Measurements are given of the absorption spectra of > 50 azomethin and indoaniline dyes in methanol and in ethanol. The effect of various substituents on the absorption maxima are indicated. Comparisons are made for some dyes between the absorption in solution and in the developed gelatin layer. C.O.C.

Syntheses of Fluorescent Brightening Agents of the Triazinylstilbene Series
A. Yabe and M. Hayashi
Kôgyô Kagaku Zasshi, **60**, 604–611 (1957): *Chem. Abs.*, **53**, 7599 (25 April 1959)

Twenty-one derivatives of disodium 4,4′-bis(2-X-4-Y-1,3,5-triazin-6-ylamino)-2,2′-stilbenedisulphonate (X and Y = same or different, OH, OCH_3, OC_2H_5, $NH_2NHC_6H_5$ or $NHC_6H_4SO_3Na$) were made by treating 4,4′-diamino-2,2′-stilbenedisulphonic acid with cyanuric chloride at 0°c. and treating with suitable reagents to replace the chlorines. The absorption spectra of aqueous solutions of these dyes in the range 220–420 mμ. are given. C.O.C.

Fluorescence and Dyeing Characteristics of Triazylstilbene Fluorescent Brightening Agents. I— Diamino and Dihydroxy Derivatives
A. Yabe and M. Hayashi
Kôgyô Kagaku Zasshi, **60**, 740–745 (1957): *Chem. Abs.*, **53**, 8635 (10 May 1957)

Exhaustion on to cotton (0·05–0·5% dye, at 20–90°c. for 5–60 min., 2–20% NaCl), fluorescence intensity and fastness to light and washing were determined of: Na 4,4′-bis(2-X-4-Y-1,3,5-triazin-6-ylamino)-2,2′-stilbenedisulphonate (X and Y = NH_2, NHC_6H_5, $NHC_6H_4SO_3Na$, OH, OCH_3 and OC_2H_5). Diamino derivatives usually show high fluorescence. The NHC_6H_5 derivative is less soluble than the $NHC_6H_4SO_3Na$ derivative. The fastness to light of the NH_2 derivative is satisfactory but that of the others is poor. Fastness to washing of diamino derivatives is generally good. The intensity of fluorescence of dihydroxy derivatives is OH < OCH_3 < OC_2H_5. Na dodecyl sulphate has negligible sensitising action on dihydroxy derivatives. The OC_2H_5 derivatives have good fastness to light. The fastness to washing of dihydroxy derivatives is poor. The 6 compounds examined appear to have no commercial possibilities. C.O.C.

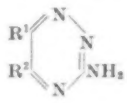
Hyperconjugation and Fluorescent Behaviour
O. Neunhoeffer, G. Alsdorf and H. Ulrich
Chem. Ber., **92**, 252–255 (1959): *Chem. Abs.*, **53**, 10187 (10 June 1959)

3-Methyl- and 3-trifluoromethyl-1,5-diphenyl-Δ^2-pyrazoline show fluorescence as a result of hyperconjugation; the 3-$(CH_3)_3C$ analogue of the first compound is not

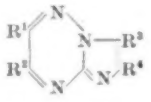
fluorescent. Solvatochromic effects observed with the first two compounds seem to be caused by simultaneous polarisation and hyperconjugation. C.O.C.

Fluorescent Pigments from 1,4,7,9-Tetrazaindene
S. Rossi and R. Trave
Chim. e Ind. (Milan), **40**, 827–830 (1958): *Chem. Abs.*, **53**, 7194 (25 April 1959)

Fluorescent pigments were obtained by reacting R^1COCOR^2 with 3-aminoguanidine and combining the resulting *as*-triazines with $R^3COCHBrR^4$. Thus aminoguanidine bicarbonate was added in small portions to R^1COCOR^2 in acetic acid. After boiling for 2–6 hr., the aminotriazine was isolated by dilution and recrystallisation from acetic acid or $HCON(CH_3)_2$. These aminotriazines are pale to deep yellow solids of high m.p. and have the formula—



When heated in solution with a bromoketone, e.g. β-$C_{10}H_7COCHBrC_6H_5$, the characteristic yellow or red of the tetrazaindene pigments quickly appeared. They have the formula—



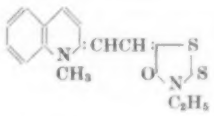
They are slightly soluble in many organic solvents, are fluorescent when solid, the fluorescence becoming much stronger in very dilute solutions. In $HCON(CH_3)_2$ their maximum absorption spectrum is in the visible range. Examples are (R^1, R^2, R^3, R^4, hue): (R^1 + R^2 =) 9,10-dihydro-9,10-phenanthro, C_6H_5, C_6H_5, rose-vermilion; (R^1 + R^2 =) 9,10-dihydro-9,10-phenanthro, H, *p*-$O_2NC_6H_4$, ochre; C_6H_5, C_6H_5, C_6H_5, C_6H_5, yellow; C_6H_5, C_6H_5, C_6H_5, α-$C_{10}H_7$, yellow; C_6H_5, C_6H_5, C_6H_5, β-$C_{10}H_7$, yellow; (R^1 + R^2 =) 9,10-dihydro-9,10-phenanthro, C_6H_5, β-$C_{10}H_7$, dark red; (R^1 + R^2 =) 2,3-dihydro-2,3-thianaphtheno, C_6H_5, C_6H_5, orange; (R^1 + R^2 =) 2,3-dihydro-2,3-indolo, C_6H_5, C_6H_5, reddish orange; (R^1 + R^2 =) 5,7-dibromo-2,3-dihydro-2,3-indolo, C_6H_5, C_6H_5, reddish orange; (R^1 + R^2 =) 1,2-dihydro-1,2-naphtho, C_6H_5, C_6H_5, reddish orange; (R^1 + R^2 =) 1,2-dihydrobenzo, C_6H_5, C_6H_5, red; (R^1 + R^2 =) 5,6-dihydro-5,6-benzo[*a*]phenazino, C_6H_5, C_6H_5, red; (R^1 + R^2 =) 3-phenyl-5,6-dihydro-5,6-benzo[*f*]quinoxalo, C_6H_5, C_6H_5, red. Those with $R^1 = R^2 = C_6H_5$ absorbed in the indigo region of the spectrum and were yellow. Consolidation of R^1 and R^2 into a single cyclic system resulted in shift of the hue to the orange–red region. The pigments are resistant to heat and can be used in inks, coloured and fluorescent varnishes, and are readily incorporated into thermoplastic resins. C.O.C.

Symmetrical and Unsymmetrical, *meso*-substituted Trimethincyanines from 2,6-Dimethylbenzo[1,2,4,5] bisthiazole
W. Ried and R. Bender
Chem. Ber., **91**, 2798–2802 (1958): *Chem. Abs.*, **53**, 8119 (10 May 1959)

The diquaternary salts of 2,6-dimethylbenzo[1,2,4,5]-bisthiazole yield *meso*-substituted sym. trimethincyanines with ortho esters and *meso*-substituted unsymmetrical trimethincyanines with heterocyclic thioethers. The absorption maxima of these dyes are shifted to longer wavelengths than those of the corresponding benzothiazole trimethincyanines. C.O.C.

Syntheses with Vinylogous Acid Halides. III— Substituted 5-Methylenerhodanines from 5-Chloromethylenerhodanines
H. Behringer, E. Dillinger, H. Suter, and K. Kohl
Chem. Ber., **91**, 2773–2783 (1958): *Chem. Abs.*, **53**, 8119 (10 May 1959)

5-Chloromethylenerhodanines, as vinylogues of acid chlorides, undergo Friedel-Crafts reaction with aromatic compounds to yield 5-arylidenerhodanines. Further reactions with active methylene compounds and with metal-organo compounds are described. Desulphurisation of the 5-arylidenerhodanines with Raney Ni yields the corresponding β-arylpropionamides. The 3-ethyl derivative of 5-chloromethylenerhodanine treated with 1-methyl-2-methylenedihydroquinone in ether with cooling yields the violet—



C.O.C.

Anthraquinone Acid Dyes
R. Shibata, N. Ishikawa, M. Suizu, T. Tanabe, and Y. Shinabe

I— Syntheses of dianilino and aminoanilino derivs.
Yūki Gōsei Kagaku Kyōkaishi, **17**, 148–153 (1959): *Chem. Abs.*, **53**, 8633 (10 May 1959)

Bluish-green to navy blue acid dyes of excellent light fastness on wool or nylon are made from 1,4-dichloroanthraquinone-7-Na sulphonate. This is (*a*) refluxed with aniline and Na_2CO_3 or (*b*) heated with aq. aniline, Na_2CO_3 and Cu_2O to give (*a*) 1,4-dianilinoanthraquinone-7-Na sulphonate (wool, nylon— bluish green) or (*b*) 1-chloro-4-anilinoanthraquinone-7-Na sulphonate (wool— bluish red). The latter heated with NH_3 and *p*-toluene sulphonamide in an autoclave gives the corresponding 1-amino compound (wool— navy blue).

II— Syntheses of dimethylanilino derivs.
Ibid., 154–158

1,4-dichloroanthraquinone-7-Na sulphonate is heated with aq. CH_3NH_2·HCl and Na_2CO_3 to give the intermediate 1-methylamino-4-chloroanthraquinone-7-Na sulphonate. Reaction with aniline and Na acetate at 183–185°c. gives 1-methylamino-4-anilinoanthraquinone-7-Na sulphonate (wool— blue black). E.T.

Disperse Dyes
K. Naiki, K. Noguchi

XIV— Synthesis of 1-amino-2-bromo-4-hydroxyanthraquinone
Yūki Gōsei Kagaku Kyōkaishi, **17**, 140–141 (1959): *Chem. Abs.*, **53**, 8633–8634 (10 May 1959)

Reddish violet needles of 1-amino-2-bromo-4-hydroxyanthraquinone, m.p. 231–232°c. from toluene, are made by (*a*) treating 1-amino-4-hydroxyanthraquinone in glacial acetic acid with bromine in glacial acetic acid followed by heating at 96–98°c. 5 hr. or (*b*) heating 1-amino-4-methoxyanthraquinone with bromine. Dyeing affinity, light and wash fastness are compared with those of 1-amino-3-bromo-4-hydroxyanthraquinone.

XV— Syntheses of 1-amino-3-methylanthraquinone and 1-amino-4-methylanthraquinone
Ibid., 142–144

1-Amino-2-methylanthraquinone in pyridine is brominated at room temperature to give 1-amino-2-methyl-4-bromoanthraquinone, m.p. 239°c. decomp. The 1-amino group is then removed to give 1-bromo-3-methylanthraquinone (m.p. 196–199°c. from acetic acid or 201°c.) by treating the amino compound in conc. H_2SO_4 with $NaNO_2$ at 0–5°c., pouring into aq. H_3PO_4 and heating at 90°c. Aminolysis gives 1-amino-3-methylanthraquinone (m.p. 194°c. from ligroin).

1-Chloro-4-methylanthraquinone is converted to 1-*p*-toluenesulphonylamino-4-methylanthraquinone (m.p. 185–186°c.) by refluxing with *p*-toluenesulphonamide, K and Cu acetates and isoamyl alcohol. This hydrolysed with conc. H_2SO_4 gives 1-amino-4-methylanthraquinone as dark red needles, m.p. 180–182°c. E.T.

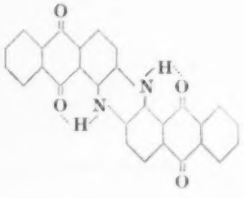
Disperse Dyes. XVI— Dyeability and Fastness of Aminoanthraquinones
K. Naiki *J. Soc. Textile Cellulose Ind., Japan*, **15**, 203–208 (March 1959)

89 Aminoanthraquinone dyes were synthesised and their dyeing and fastness properties examined. The relationship between structure and dyeing properties is discussed in terms of *a*/*o* (inorganic property/organic property) and the molecular size. *a*/*o* is calculated by use of Fujita's conceptional diagram (*Chem. Abs.*, **52**, 231 1958)). *a*/*o* is equivalent to hydrophilicity/hydrophobicity. The average *a*/*o* of dyes of good affinity for acetate is about 1·25 and this is consistent with that of

secondary cellulose acetate. Dyes capable of diffusing into cellulose acetate in normal dyeing have molecular size < 13 A. The fastness to light decreases in the following order: 1-amino-4-hydroxyanthraquinones > 1,4-diaminoanthraquinones > 1,4,5-triamino-8-hydroxyanthraquinones > 1-aminoanthraquinones > 1,4,5,8-tetraaminoanthraquinones. An electron attractive *N*-substituent, e.g. a 2-chloroethoxycarbonyl group, increases the fastness to light and an electron donating group, e.g. 2-hydroxyethyl, decreases it. The *a/o* of dyes of poor fastness to washing is > 1·25 and their molecular size is < about 11·0 A. C.O.C.

Structure of Indanthrone (C.I. 69800)
J. Weinstein and C. Merrit
J. Amer. Chem. Soc., **81**, 3759–3763 (20 July 1959)

Studies of the ultraviolet, visible and infrared spectra of indanthrone and related compounds indicate that in the solid state and in inert solvents indanthrone exists primarily in a keto form.



C.O.C.

Catalytic Action of Quinones in the Oxidation of 4-Methyl*meso*benzanthrone to *meso*Benzanthrone-4-carboxylic Acid
W. Bradley and K. H. Shah
J.C.S., 1908–1909 (May 1959)

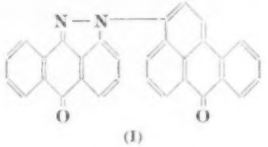
The catalytic effects of anthraquinone, 1,4,5,8-tetrachloroanthraquinone, 1,2-benzanthraquinone, anthanthrone, 1′,2′-6′,7′-dibenzopyrene-7,14-quinone, and flavanthrone are compared for promoting the oxidation of 4-methyl*meso*benzanthrone to *meso*benzanthrone-4-carboxylic acid by nitrobenzene and barium hydroxide and a trace of copper sulphate. Of these quinones the most effective by far was 1,2-benzanthraquinone. No oxidation was observed when nitrobenzene was replaced by anisole. H.H.H.

Dyes from Benzoyl-3-azabenzanthrone by Alkali Fusion and from Benzoyl-1-bromobenzoyl-3-azabenzanthrone by Alkali Fusion after Treatment with Sodium Disulphide
M. Yokote
Kōgyō Kagaku Zasshi, **60**, 1045–1048 (1957): *Chem. Abs.*, **53**, 131–148 (25 July 1959)

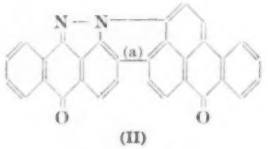
Benzoyl-3-azabenzanthrone (I) (0·6 g.) was fused for 1 hr. at 110–120°c. with ethanol (9·5 c.c.) and KOH (6·2 g.), added to water, oxidised with air and dissolved in ethanol to yield crude 2,2′-dibenzanthronyl (II). This (0·268 g.) was fused with KOH (6·018 g.) and K acetate (0·697 g.) at 280°c. for 1 hr., poured into water, oxidised with air, extracted with aqueous alkali, HCl, ethanol, acetic acid, chlorobenzene and purified by reducing to the leuco compound to yield isoviolanthrone A (III) (0·148 g.). Alkali fusion of I successively at 110–270°c. gave III in higher yield. Benzoyl-1-bromo-3-azabenzanthrone (0·37 g.) was treated for 3 hr. with Na_2S_2 (3) and S (0·41) at 150–160°c., washed with boiling water and hot ethanol and dried to give a yellowish brown product (0·244 g.), presumably benzoyl-1(or 1′)-thiadibenzoanthronyl. This (0·155) was fused for 1 hr. with KOH (0·99) at 140°c., washed with water, oxidised with air, washed with dilute alkali, water, hot acetic acid, and hot chlorobenzene to yield III (0·117 g.). C.O.C.

Formation of Quinones by Union of Ketones. Structure of Indanthren Navy Blue R (C.I. 70500)
W. Bradley and K. H. Shah
J.C.S., 1902–1908 (May 1959)

The condensation of 1,9-pyrazoloanthrone (III) with 3-bromo*meso*benzanthrone, which is found to be much improved when III is used as the potassium salt and the reaction carried out in nitrobenzene, gives 1′-(*meso*benzanthron-3-yl) pyrazolo (5′,4′,3′–1,13,9) anthrone (I), which, when heated with NaOH in pentyl alcohol at 110–120°c., is converted into Indanthren Navy Blue R (II); although sodium anilide is more active it produces degradation and substitution even at 10°c. The constitution of I is now confirmed by a series of reactions which are described. The main uncertainty in the assigned structure of II is whether the bond (*a*) really exists, but cyclisation has been verified by a study of the union of *meso*benzanthrone with 1,9-pyrazoloanthrone in the presence of bases, and so the structure of II is confirmed. The course of the reactions is shown to be ionic in character.



(I)



(II)

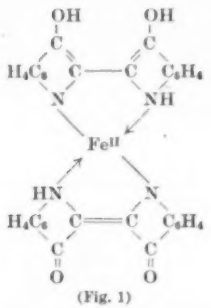
H.H.H.

NO-Indigo. Constitution of the Glycosine Dye of Radzisewski and the Benzimidazole Dye of Niementowski
R. Kuhn and W. Blau *Ann.*, **615**, 99–107 (1958): *Chem. Abs.*, **53**, 9191 (25 May 1959)

Radzisewski's blue black dye (*Ber.*, **17**, 1290 (1884)) is shown to be 2,2′-diimidazyl 3,3′-dioxide and Niementowski's dye (*Chem. Abs.*, **5**, 502 (1911)) to be 2,2′-dibenzodiimidazyl 3,3′-dioxide. The latter has two NO groups in place of the two CO groups of indigo and is called NO-indigo. C.O.C.

Model Oxygen-carrying Compounds and the Iron-Bisindigo Complex
L. F. Larkworthy and R. S. Nyholm
Nature, **183**, 1377–1380 (16 May 1959)

Some of the relevant aspects of the problem of the ability of haemoglobin to combine reversibly with molecular oxygen without itself undergoing oxidation, also the closely related problems of the orientation of the oxygen mol. in oxyhaemoglobin, and of those iron and cobalt chelate complexes which can combine reversibly with oxygen to yield cryst. cpd. are discussed, and a study of the chemistry of the iron–bisindigo complex is reported. Artificial oxygen carriers appear to be restricted to the cpd. of Co and Fe, and the μ-oxochlororhenate ion $(Re_2OCl_{10})^{4-}$. Most of the Co cpd. suffer from the disadvantage, as model cpd., that the reversibly combined oxygen mol. is believed to form a bridge between the Co atoms in two adjacent mol.; in effect, one oxygen atom is reversibly held by each Co atom. The iron–bisindigo complex (I), first prepared by Kunz and Kress (*Chem. Ber.*, **60**, 367 (1927)) bears at least a superficial resemblance to haemoglobin, and is now found to behave as a normal paramagnetic cpd. ($\theta = -25°$K.) with magnetic moment 5·4 B.M., corresponding to that of ferrous iron in a spin-free complex. A soln. of I in pyridine absorbs one oxygen mol. for each iron atom with colour change from red to green, and apparent reversal when pressure over the soln. is reduced, or if the green soln. is allowed to stand under nitrogen. Fig. 1 depicts a structure consistent with the infrared spectra of I, but arrangements in which the iron atom is co-ordinated to one oxygen and one nitrogen atom of the indigo residue are also feasible. It has now been found, however, that purified ferrous–bisindigo is not capable of reversible combination with oxygen in the absence of iron pentacarbonyl, which is one of the reactants for the preparation of I and so was present as an impurity in all the earlier specimens. In consequence, I cannot be used as a model for haemoglobin.



(Fig. 1)

H.H.H.

Preparation and Properties of the Sulphonic Acids of Copper Phthalocyanine (C.I. 74160)

A. A. Spryskov and A. I. Kobenin

Trudy Ivanovsk. Khim.-Tekhnol. Inst., (5), 196–201 (1956): *Referat. Zhur., Khim.*, Abstr. No. 24440 (1957): *Chem. Abs.*, **53**, 13601 (25 July 1959)

The light absorptions of the mono-, di-, and trisulphonic acids of C.I. 74160 are almost identical. The Na disulphonate exhausts best on to cotton. Purified C.I. 74160 was treated with 20% fuming H_2SO_4 at 65°c. for 3 hr., poured on to ice, filtered off, washed with 5% HCl, dissolved in boiling 1% NaOH and filtered. The filtrate was acidified with HCl, stood for 10–15 hr. and then the monosulphonic acid of C.I. 74160 filtered off. C.I. 74160 (30 g.) was treated with $ClSO_3H$ (100 ml.) for 5 hr. at 100°c., poured on to ice, filtered off, dissolved in alkali, filtered and the disulphonic acid of C.I. 74160 separated by acidifying the filtrate with HCl and purified by boiling 3–4 times with dil. HCl. The trisulphonic acid was obtained by analogous sulphonation at 140°c. C.O.C.

Reprecipitation of Copper Phthalocyanine (C.I. 74160)

V. F. Borodkin, K. V. Usacheva, and A. I. Dormidontova

Izvest. Vysshykh Ucheb. Zavedenii, Khim. i Khim. Tekhnol., (5), 131–135 (1958): *Chem. Abs.*, **53**, 10797 (10 June 1959)

Cu phthalocyanine (C.I. 74160) is insoluble in 58% H_2SO_4 at 20°c. even in presence of 15-fold excess of acid and prolonged stirring, but swells and forms microsuspensions. In 96% H_2SO_4 (5–7-fold excess) the sulphate is formed and becomes soluble when the acid is increased to 12-fold excess. Increase in the temperature permits solution of C.I. 74160 in less H_2SO_4, e.g. at 100°c. it dissolves in 7- and 5-fold excess of 85 and 96% H_2SO_4 respectively. The pigment obtained by reprecipitation from 85% H_2SO_4 is a purer blue than that from 96% H_2SO_4 and is greener than that of the typical sample obtained with castor oil. Treatment of C.I. 74160 at 10, 30, and 100°c. in 85% H_2SO_4 yields a pigment of purer blue than in 96% H_2SO_4. At $>$ 100°c. the solution of C.I. 74160, independent of the strength of the acid, gives a pigment with unsatisfactory colour coefficients. Preliminary dilution with water of the acid solution with subsequent separation of the pigment from the crystals of C.I. 74160 sulphate did not improve the purity of the colour. Use of castor oil during the reprecipitation of C.I. 74160 makes the process difficult and does not guarantee a pigment of good colour. Of a few compounds studied as dispersing agents the most effective was triethanolamine soap (5–10% on the weight of pure C.I. 74160). Optimal conditions for precipitation of C.I. 74160 are: solution of technical C.I. 74160 in 96% H_2SO_4 (1 : 10 pigment : acid) at $\geq$ 25°c. with subsequent separation through a sprayer in 30% H_2SO_4 containing triethanolamine soap. C.O.C.

FD&C Yellow Nos. 1, 2, 3, and 4; Ext. D&C Yellows Nos. 7, 8, 9, and 10

Federal Register, **24**, 883–884 (6 Feb. 1959): *Chem. Abs.*, **53**, 12505 (10 July 1959)

FD&C Yellow 1, 2, 3, and 4 are deleted from the list of dyes certifiable under the U.S. Federal Food, Drug, and Cosmetic Act. They are added, as Ext. D&C Yellow Nos. 7, 8, 9, and 10 to the list of dyes certifiable for use in externally applied drugs and cosmetics. C.O.C.

Colorants for Synthetic Sake. I— Colour Tone and Fading of Artificial Colorants

Y. Nishiwaki

Hakkó Kyókaisha, **16**, 391: *Chem. Abs.*, **53**, 9563 (25 May 1959)

The absorption spectra of Caramel (C.I. Natural Brown 10 and Melanin (C.I. Natural Brown 9) are very similar to that of brewed sake. Dilute solutions of Caramel and Melanin fade in sunlight or ultraviolet radiation, Caramel fading somewhat quicker than Melanin. C.O.C.

Crystallised Natural Chlorophylls *a* and *b*

A. Stoll and E. Wiedemann

Helv. chim. Acta, **42**, 679–683 (4 May 1959)

After several decades of endeavour (briefly summarised) the goal of crystallising the natural chlorophylls *a* and *b* has been attained by chromatographic separation on powdered sugar, directions for which are given. The crystalline substances so obtained meet all criteria for pure chlorophylls *a* and *b*, and their infrared spectra are reported for the first time. H.H.H.

Chlorophyll Compounds with Bivalent Cobalt and Zinc

N. V. Vostrilova and V. I. Dulova

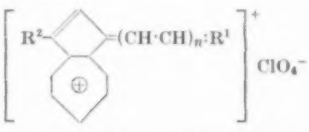
Uzbek. Khim. Zhur., Akad. Nauk S.S.R., (1), 69–79 (1958): *Chem. Abs.*, **53**, 9384 (25 May 1959)

Azulenestyryl Dyes

E. J. Poppe and W. Treibs

Naturwissenschaften, **45**, 517 (1958): *Chem. Abs.*, **53**, 7599 (25 April 1959)

Azulene condensed in presence of $POCl_3$ with 2-formylmethylene compounds containing heterocyclic N gave dyes of formula—



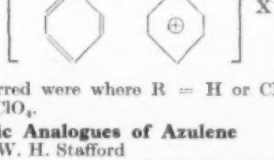
e.g (compound from which R^1 is derived, R^2, and *n*); *N*-ethylbenzothiazoline, H, 1; 5,6-dimethyl-*N*-ethylbenzothiazoline, H, 1; *N*-ethylbenzoselenazoline, H, 1; 1,3,3-trimethylindoline, H, 1; 1,3,3,5-tetramethylindoline, H, 1; *N*-ethylbenzothiazoline, CH_3CONH, 1; *N*-ethylbenzothiazoline, H, 2 C.O.C.

Nitrogen-free Azulenemonomethin Dyes

G. Bach, E. J. Poppe, and W. Treibs

Naturwissenschaften, **45**, 517 (1958): *Chem. Abs.*, **53**, 7600 (25 April 1959)

Reaction of an azulene with C_1-orthoformate or a thioformic ester in presence of $POCl_3$ gave unstable monomethin dyes of formula—



Those preferred were where R = H or CH_2CONH and X = Cl or ClO_4. C.O.C.

Heterocyclic Analogues of Azulene

M. Los and W. H. Stafford

J.C.S., 1680–1685 (May 1959)

The preparation and properties of 4-methyl-2-phenyl- and 1,2-diphenyl-β-quinindene and of 2-phenyl- and 1,2-diphenyl-benzo[*b*]*cyclo*penta[*e*]pyran are described. These cpd. are analogues of 5,6-benzazulene (I) and contain nitrogen and oxygen respectively in the central benzene ring; they are highly coloured (violet or blue) and behave as anhydro salts. Their spectra are discussed with respect to that of I. H.H.H.

Litmus and Related Dyes

T. Hirakata

Ann. Rept. Fac. Pharm. Tokushima Univ., **4**, 5–10 (1955): *Chem. Abs.*, **53**, 6188 (10 April 1959)

Study of the relationship between the structure of the raw material and the colour of the dye obtained from it was made for litmus dyes (I) and lacmoid dyes (II) prepared from alkylresorcinol derivatives. It is shown that (I) I can be prepared from orcinol derivatives but

not from resorcinol; (2) II was less coloured than I; (3) coloration of both dyes was decreased by introduction of CH_3 groups; (4) introduction of aldehyde groups intensified the colour of I and diminished that of II. C.O.C.

Orcein Dyes

IV— Craig Distribution and Distribution Chromatography

H. Musso and H. Beecken

Chem. Ber., **90**, 1808–1814 (1957)

The uniformity of the 7 crystallised main components of orcein was ascertained by Craig distribution. Hydroxy orceins are very sensitive to air oxidation in solution at pH 11–12. Comparison of distribution chromatography and Craig distribution proved a possible separation effect of 200–600 theoretical distribution steps for 1 cm. cellulose column.

V— Infrared Ultraviolet Spectra of Hydroxy- and Amino-substituted Phenoxazones

H. Musso and H. G. Matthies *Ibid.*, 1814–1827:
Chem. Abs., **53**, 9674 (25 May 1959)

The spectra of 2-phenoxazone and a number of its hydroxy and amino substituted derivatives have been studied to obtain information on the chromophoric skeleton of all orcein components.

IX— Fluorescence of Orcein Dyes

H. Musso and H. G. Matthies

Naturwissenschaften, **46**, 15 (1959):
Chem. Abs., **53**, 11842 (25 June 1959)

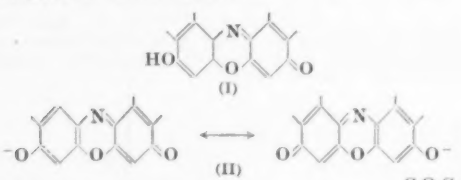
1,9-Dimethyl-7-amino- (I) and 1,9-dimethyl-7-hydroxy-3-phenoxazones (II) substituted in the 8- or 2,8-positions by 2,4,6-$(HO)_2C_6H_2CH_3$ (III) fluoresce in methanol at the temperature of liquid air. The fluorescence is quenched by warming but I and II in ethanol fluoresce even at room temperature. The quenching of the fluorescence of the substituted I and II is attributed to presence of the bulky III group, which either absorbs the excitation energy or dissipates it by some unknown mechanism. C.O.C.

X— Light Absorption and Chromophore of Litmus, C.I. Natural Red 28

H. Musso and C. Rahtjen

Chem. Ber., **92**, 751–753 (1959):
Chem. Abs., **53**, 14105 (10 July 1959)

Spectroscopic investigation has shown that the colour change of litmus with acids and alkalies depends on the conversion of a red substituted 7-hydroxy-2-phenoxazone of structure I to the anion II—



(I)

(II)

C.O.C.

Test with C.I. Food Orange 2

A. Sporn and J. Heilpern *Igiena*, **7**, 235–243 (1958):
Chem. Abs., **53**, 7447 (25 April 1959)

Tests with rats and mice show that Orange GGN even in very large doses has not the slightest harmful effect. C.O.C.

Colour Certification in the U.S.A.

Federal Register, **24**, 3851–3865 (14 May 1959)

Republication and recodification of regulations including definitions and specifications for coal-tar dyes that can be used under the U.S. Federal Food, Drug, and Cosmetic Act for foods, drugs and, cosmetics; drugs and cosmetics; and in externally-applied drugs and cosmetics. C.O.C.

Natural Dyes and their Use in Dyeing Foods

A. Janković *Teknika* (Belgrade), **14**, 121–125 (1959):
Chem. Abs., **53**, 11683 (25 June 1959)

Review, 15 references. C.O.C.

Constitution of the Colouring Matter of Spruce Bark, a New Class of Condensable Tannin

H. Endres *Qualitas Plant et Materiae Vegetabiles*, **5**, 367–374 (1959):
Chem. Abs., **53**, 14234 (10 July 1959)

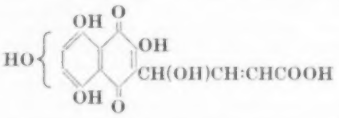
The cambium layer of spruce bark containing 15–17% of tanning material was extracted with ethyl acetate and ethanol under non-oxidising conditions; this removed 30% and 20% respectively of the tannins. Enzymic hydrolysis of the ethyl acetate extract followed by fractionation on a polyamide column led to the isolation of two crystalline phenolic compounds. The main product (45% of the aglucone mixture) was 2,3′,4′,5,6-pentahydroxy-3,4-tetramethylstilbene and has been named piceatannol. The second phenol (15% of the mixture) is a piceatannol derivative containing a saturated stilbene double bond. Piceatannol exists in the cambium as mono- and diglucosides. Direct chromatography of the ethyl acetate extract in a silica gel–cellulose column gives the diglucoside in which glucose is attached to the 5- and 3′-positions. The ethanol extract contains 50% of a monoglucoside of piceatannol. Two monoglucosides of dihydropiceatannol have been isolated in a 10% yield. Piceatannol formed a water-soluble condensate when treated with the phenol-oxides present in the cambium and did the same with oxidising agents. C.O.C.

Derivatives of Naphthaquinones. XIV— Pigments from Sea Urchins. 9. Spinochrome M

C. Kuroda and M. Okajima

Proc. Japan. Acad., **34**, 616–618 (1958):
Chem. Abs., **53**, 14064 (10 July 1959)

The yellow pigment Spinochrome M has the formula—



C.O.C.

Ferric Red by a Topochemical Method

A. Krause and M. Kranz

Przemysl Chem., **12**, 312–315 (1956):
Chem. Abs., **53**, 7619 (25 April 1959)

Dehydration by topochemical treatment for 30–180 min. at 150–180°c. and 5–10 atm. of the yellow product obtained by heating $Fe_2(SO_4)_3 \cdot 9H_2O$ with dil. NaOH, yields a series of red and pink pigments. C.O.C.

Precipitation of Yellow and Brown Hydrated Ferric Oxide (C.I. 77492). XI— Electron Diffraction Study of Various Hydrated Ferric Oxides

S. R. Gupta and S. Ghosh *Proc. Natl. Acad. Sci. India*, Sect. A, **23**, Pt. 1, 44–47 (1954):
Chem. Abs., **53**, 7722 (10 May 1959)

Adding NaOH to aq. $FeCl_3$ in presence of SO_4^{--} or $Cr_2O_7^{--}$ immediately gave a brown precipitate followed by slow precipitation of a yellow hydrated ferric oxide free of Cl^-. The brown and yellow hydrated Fe_2O_3 differ in chemical and physical properties from *a* brown hydrated Fe_2O_3. Electron diffraction patterns showed the yellow material is a different form from those previously described. C.O.C.

Synthesis of Ultramarine (C.I. 7707) from Anzhero Kaolin

V. V. Boldyrev

Trudy Tomsk. Univ., (126), 70–72 (1954):
Referat. Zhur., Khim., Abstr. No. 17832 (1957):
Chem. Abs., **53**, 11856 (25 June 1959)

Kaolin from the Yaisk and Anzhero–Sudyhensk deposits (34·5–40·0%), soda (35–38), S (17·5–24), coal (3·5–7·5), and borax (5–7) are calcined by the soda method to produce blue ultramarine. The kaolin must be first calcined for 3–4 hr. at 500°c. to remove its moisture content which would otherwise interfere with the formation of ultramarine. C.O.C.

Structure of Particles of Carbon Black (C.I. Pigment Black 6 and 7)

H. P. Boehm

Z. anorg. u. allgem. Chem., **297**, 315–322 (1958):
Chem. Abs., **53**, 7711 (10 May 1959)

Electron-microscopic examination shows that the crystallites in the spheroidal particles of Thermax (a Furnace Black) are oriented with their basal planes parallel to the surface. The shell-like structure arises from deposition of carbon layers on crystal nuclei in the gaseous phase rather than on other surfaces present at the time of decomposition of gaseous hydrocarbon. Particles of Thermax graphited at 2000–3000°c. consist of several

pyramidal graphite crystals whose apexes are directed to the centre; the faces of the resultant polyhedra are the basal surfaces of the graphite crystals. C.O.C.

Organic Nature of Carbon Black (C.I. Pigment Black 6 and 7) Surfaces. II—Quinones and Hydroquinones by Coulometry at Controlled Potential

H. V. Drushel and J. V. Hallum

J. Phys. Chem., **62**, 1502–1505 (1958): *Chem. Abs.*, **53**, 6907 (25 April 1959)

Describes a quantitative method for determining quinone O and hydroquinone OH on the surface of Carbon Black by coulometric reduction or oxidation at controlled potential. Among a group of Carbon Blacks used in the rubber industry the quinone O varied from 0·002–0·255% and the hydroquinone OH from nil to 0·470%. C.O.C.

Unpaired Electrons in Carbon Black (C.I. Pigment Black 6 and 7)

R. L. Collins, M. D. Bell, and G. Kraus

J. Appl. Phys., **30**, 56–62 (1959): *Chem. Abs.*, **53**, 10973 (25 June 1959)

An appreciable concentration of unpaired electrons were found in colloidal Carbon Blacks by application of electron spin resonance spectroscopy. The unpaired electrons in a typical channel black, Spheron 6, were studied by noting changes in the electron spin resonance spectrum produced by heating to 3000°c. and by various degrees of evacuation of the sample. Results indicated that the unpaired electrons in the original material were mobile π electrons stabilised by condensed aromatic ring structures. Their concentration decreases slowly to 750°c. and then declines more quickly and vanishes at > 1400°c. A different species of unpaired electrons arises at 1400°c. These seem to be immobile σ electrons resulting from broken C–C bonds at lattice defects. Unpaired electrons present in several commercial Carbon Blacks on examination seem also to be π electrons. A tentative theory of the origin of the unpaired electrons is given. C.O.C.

New Cobalt Pigments

O. Schmitz-DuMont

Forschungsber. Wirtsch. -u. Verkehrsministeriums Nordrhein-Westfalen No. 193, 37 pp. (1955): *Chem. Abs.*, **53**, 10796 (10 June 1959)

An investigation of the co-ordination, lattice parameter, and $Co^{++} \longleftrightarrow O^{--}$ coupling on the absorption spectra of oxidic co-ordination structures of Co^{++}. Octahedral co-ordination was achieved by isomorphous Co^{++} exchange in MgO and in inverse spinel $MgIn_2O_4$ lattices. The absorption spectra of $Ba(CoPO_4)_2$ and $Co_3(PO_4)_2 \cdot 8H_2O$ (C.I. 77360) were investigated. Tetrahedral co-ordination of Co^{++} produces the spectra of the normal spinels $CoAl_2O_4$ (C.I. 77346) and $CoGa_2O_4$. Preparation and X-ray diffraction studies were made of $CoAl_{2-n}Ga_nO_4$ (I), $CoAl_{2-n}In_nO_4$ (II), $CoGa_{2-n}In_nO_4$ (III) and $Ba(Zn_{1-n}Co_n)PO_4$ IV. Shift of the absorption to smaller wavelengths in blue type I indicates the influence of the lattice forces in the absorption spectrum. $CoAl_2O_4$ and $CoGa_2O_4$ form a continuous series of mixed crystals with linearly increasing lattice parameters. In $CoAl_2O_4$ only 15 atoms % Al^{+++} are exchangeable by In^{+++}. Compounds containing higher In contents consist of green II and $CoO \cdot In_2O_3$. Of Ga 30 atoms % are replaceable by In in $CoGaO_4$. A tetragonal phase was observed between 30–70 atoms % Ga. Introduction of Co^{++} in Mg_2SnO_4 occurs on tetrahedral sites. Co^{++} ions in Zn_2TiO_4 are distributed over octahedral sites. Substitution of Co^{++} in Zn_2SnO_4 and Mg_2TiO_4 occurs in tetrahedral and octahedral sites. The abnormal absorption spectrum and magnetic behaviour of $Co_{2-x}Mg_{1+x}TiO_4$ are caused by the coupling effect. $Ba(ZnPO_4)_2$ (V) was prepared by mixing solutions containing 0·01 mole Ba^{++}, 0·02 mole Zn^{++} and 0·03 mole H_3PO_4 and neutralising with 20% NaOH to pH 5·8–6·0. It is a white pigment crystallising in needles. Blue, light pink, and violet IV compounds are obtained by successive replacement of Zn^{++} by Co^{++} solution. The X-ray diagrams and absorption spectra of type-IV compounds are given. C.O.C.

Electronic Spectra of Titanium Dioxide, C.I. Pigment White 6

H. Bevan, S. V. Dawes, and R. A. Ford

Spectrochim. Acta, **13**, 43–49 (1958): *Chem. Abs.*, **53**, 6778 (25 April 1959)

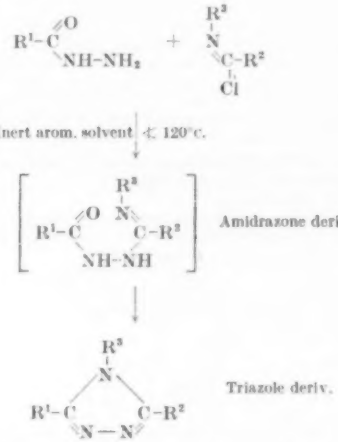
The absorption spectra of thin evaporated films of anatase and rutile have been recorded between 20,000 and 50,000 cm.$^{-1}$. The intense absorption in this region is attributed to an exciton transition arising from transfer of an electron from an O to a Ti atom. The number and symmetry properties of the excited states associated with this configuration are discussed. Splitting of the exciton band observed in both anatase and rutile is related to the distortion of the TiO_6 octahedra in these crystals. C.O.C.

PATENTS

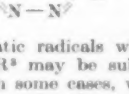
Triazole and Amidrazone Anthraquinone Vat Dye Intermediates

American Cyanamid *USP* 2,884,424

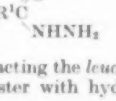
Preparation of these intermediates takes place via the following reaction—



(R^1, R^2, R^3 = aromatic radicals where < 1 is anthraquinonoid; R^1, R^2, R^3 may be subst. with Hal, NH_2, alkoxy, Alk etc.). In some cases, where reaction ceases at the amidrazone stage, ring closure is effected separately by heating with $SOCl_2$ or chlorosulphonic acid. The present patent also states that an improvement on existing methods of making the anthraquinone carbohydrazides—



above, is effected by reacting the *leuco*-deriv. of an anthraquinone-2-carboxylic ester with hydrazine. The chloroimides—

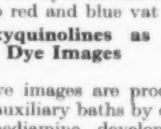


above, are prepared by reaction between a monosubstituted amide and PCl_5 or thionyl chloride. The unsymmetrical 1,2,4-triazoles obtainable by the above reactions are either vat dyes themselves or may be used to prepare yellow to red and blue vat dyes. E.T.

5-Amino-8-hydroxyquinolines as Coupling Components for Azine Dye Images

General Aniline *USP* 2,886,436

Magenta azine dye images are produced directly and without use of any auxiliary baths by colour development with a *p*-phenylenediamine developer if the colour former used is of formula—



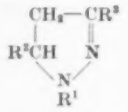
(W = Alk, hydroxyalkyl, carboxyalkyl, sulphoalkyl, or Ar of the benzene series, alkoxyphenyl, carboxy- or sulphophenyl; Y = H, acyl or sulphonyl; A = H, carboxyl

or carbamino; B = H, Alk or aromatic radical of the benzene series; C = H, carboxy, Alk or aromatic radical of the benzene series), e.g. 3-tetradecanoylamido-4-methyl-5-[8′-hydroxy-6′-(*N*-methylbenzenesulphonamido)cinchoninoyl]-3-amidobenzene sulphonic acid. C.O.C.

Fluorescent Compounds for making Photographic Material Distinguishable in Ultraviolet Radiation

Società per Azioni Ferrania *BP* 816,026

Compounds of formula—

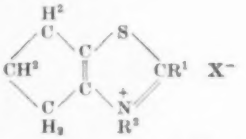


(R^1, R^2 and R^3 = aromatic radicals at least one of which is attached to the pyrazoline ring by a methin chain $(CH{:}CH)_n$ (n = 1 or 2)) when incorporated in a cellulose derivative used as a photographic film base fluoresce strongly under ultraviolet radiation. Thus 1-(2-benzothiazolyl)-3-styryl-5-phenyl pyrazoline displays an intense greenish fluorescence in ultraviolet radiation. C.O.C.

2-Methyl-5,6-dihydro-4-cyclopentathiazoles as Intermediates for Cyanine Dyes

Sperry Rand Corpn. *USP* 2,882,160

The title compounds are useful for preparing a wide range of organic dyes. They are first converted into quaternary salts of formula—

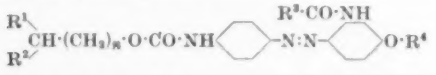


(R^1 = CH_3, C_2H_5 or *n*-propyl; R^2 = Alk or aralkyl; X = anion) and then treated with a second intermediate in presence of an acid binding agent. This treatment with *α*-halogenoquinoline quaternary salts yields pseudocyanine dyes, while symmetrical carbocyanine dyes are obtained if an ester of an ortho acid is used as the second intermediate. Other combinations are given. C.O.C.

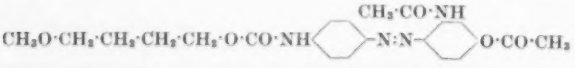
Yellow Monoazo Disperse Dyes

S *BP* 816,308

Monoazo dyes of general formula—



(n = integer from 0 to 5; R^1 = H or Alk of 1–4 C; R^2 = H, OH, OAlk, $NAlk_2$, or Alk of 1–4 C; R^3 = Alk of 1–4 C; R^4 = acyl of 2–4 C, CH_3, C_2H_5, or C_2H_4OH) dye cellulose acetate yellow from aq. dispersion or may be incorporated in the spinning solutions of cellulose acetate and other thermoplastic fibre-forming polymers. Thus the monoazo compound in which R^1 = H, R^2 = OCH_3, R^3 = CH_3, R^4 = H, n = 3 is stirred in pyridine with acetyl chloride to give—

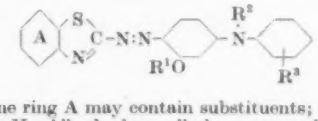


which dyes cellulose acetate and nylon yellow of high light fastness. Ninety examples are given. E.S.

Metallisable Monoazo Disperse Dyes

Eastman Kodak Co. *BP* 816,396

2-Aminobenzthiazoles are diazotised and coupled with suitable diphenylamine derivatives to give monoazo compounds—



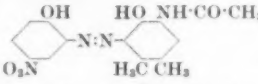
(the benzene ring A may contain substituents; R^1 = H or Alk; R^2 = H, Alk, hydroxyalkyl, or cyanoalkyl; R^3 = H, OH, OAlk, or Cl) which may be metallised, e.g. after applying as a disperse dye to cellulose acetate. Thus 2-amino-6-methylsulphonylbenzthiazole is diazotised and coupled with 3-hydroxydiphenylamine, to give the monoazo compound which dyes cellulose acetate red, converted to violet by padding with $Ni(SCN)_2$ followed by steaming.

Preparations of ten suitable 6-substituted-2-aminobenzthiazole derivatives are given in some detail. E.S.

Brown Metal (Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

Gy *BP* 815,790

Metal complexes containing 1 atom of Co or Cr and 2 mol. of monoazo compounds obtained by coupling a diazotised 2-amino-4-nitrophenol (which may be substituted in the 6-position by Hal, Alk, etc.) with a *p*-cresol derivative having CH_3 in the 2(or 5)-position and an acylamino group in the 5(or 2)-position, dye wool and nylon brown from a neutral or weakly acid bath. Thus diazotised 2-amino-4-nitrophenol is coupled with 2-acetamido-4,5-dimethylphenol giving—



Boiling with aq. sodium chromosalicylate gives the Cr-complex. E.S.

Production of Azo Pigments in Finely-divided Form

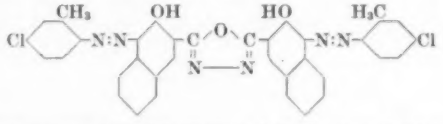
General Aniline *BP* 816,585

Insoluble azo pigments are produced in a finely-divided form suitable for incorporation in fibre spinning solutions by carrying out the coupling operation in presence of 25 to 175 g. of an anionic surface-active agent per g. mol. of pigment. Thus to a soln. of 3-hydroxy-2-naphtho-5′-chloro-2′,4′-dimethoxyanilide in aq. NaOH is added sodium acetate (6 g.) and Tamol NNO (formaldehyde naphthalene sulphonate) (5 g.). This soln. is then run into a diazo soln. prepared from 2-aminoanisole-5-sulphondiethylamide (12·9 g.) and containing Emulphor ON (polyoxyethylene ether of a higher fatty alcohol) (1 g.). After stirring for 1 hr. cold, the charge is boiled for 1 hr. to give a filterable product. Mixing the filter-cake with 10% Tamol NNO and passing over a roller mill gives the finished product containing particles 99% of which are $< 1\ \mu$. in diameter. E.S.

Disazo Pigments Having Oxdiazole Rings

Ciba *BP* 816,749

Disazo compounds free of solubilising groups but containing oxdiazole rings, e.g.—

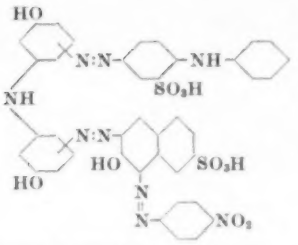


are pigments of good fastness to light, heat, and solvents. This example is prepared by treating the monoazo compound 2-amino-5-chlorotoluene→3-hydroxy-2-naphthoic acid suspended in chlorobenzene with thionyl chloride, thereby converting the COOH group into COCl. Two mol. of the acid chloride are condensed with 1 mol. of hydrazine by boiling in chlorobenzene, and ring closure is then effected by adding thionyl chloride and boiling further. The product colours synthetic polymers, e.g. polyvinyl chloride red of good fastness to migration and light. E.S.

Brown Tris- and Tetrakis-Azo Dyes for Leather

FH *BP* 815,737

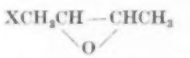
Tris- and tetrakis-azo dyes derived from 3,3′-dihydroxydiphenylamine (I) by e.g. coupling it first with 1 mol. of a suitable diazotised aminonaphtholsulphonic acid, and then coupling the monoazo compound so produced with 2 mol. of diazo compounds of the benzene or naphthalene series containing at least one SO_3H group, give browns on leathers of various tannages. Thus 1 mol. of diazotised 2-amino-3-naphthol-6-sulphonic acid is coupled with I. A diazo suspension prepared from 4-aminodiphenylamine-2-sulphonic acid is then added, followed by aq. NaOH to maintain a pH value of 10. Finally diazotised *p*-nitroaniline is run in together with more aq. NaOH, giving—



which dyes leather deep brown. E.S.

Dibenzanthrone Vat Dyes
DuP *USP* 2,888,463

Vat dyes of outstanding brightness and fastness to water-spotting and ironing are made by etherifying 16,17-dihydroxybenzanthrone (Bz-2, Bz-2′-dihydroxybenzanthrone) with 2–5 mol. of (*a*) cyclohexanone, cyclopentanone or aliph. ketones of formula XCH_2COCH_2Z and $XCH_2COCHYCH_3$ (X, Y = H, Cl or Br; Z = Cl or Br), or (*b*) dimethyl, diethyl or ethylene ketals corresponding to the above ketones, or (*c*) epoxides of formula—



Reaction takes place in 2–72 hr. in 10–20 parts 85–100% H_2SO_4 at 30–60°c. and ≯ 75°c. Separation is effected by drowning the melt into ice/water and filtering. The method gives products which differ slightly from those obtained by the process of *USP* 2,218,663 but the present method, by eliminating the use of organic solvents and expensive alkylating agents, is more economical and easier to control. Reference is also made to vat dyes made by the method of *BP* 206,638 (J.S.D.C., **40**, 53 (1924)). E.T.

Dioxy-Nitro-Arylaminoanthraquinone Disperse Dyes
Ciba *BP* 815,746

Blue dyes for cellulose esters, nylon, and Terylene having good fastness to light and gas fumes are made by condensing mixed dioxy-dinitro-anthraquinones (containing ≮ 40% of the isomers 1,5-dioxy-4,8-dinitro-anthraquinone and 1,8-dioxy-4,5-dinitro-anthraquinone) with an arylamine and ≯ 5% water at 100–150°c. The above anthraquinone starting material may be obtained by (*a*) dinitrating anthraquinone to give mixed 1,5- and 1,8-dinitro-anthraquinones which also contain other α- and β-nitro-derivatives; (*b*) replacing the NO_2 groups by OCH_3 or phenoxy; (*c*) dinitrating the products and (*d*) hydrolysing the OCH_3 or phenoxy groups. The arylamine may be aniline unsubst. or subst. by Hal, Alk or alkoxy of ≯ 4 C, e.g. $H_2NC_6H_{5-n}(CH_2)_{n-1}$ where n ≯ 3. The dyeing properties of the products are improved by acid pasting. Their general properties are claimed to be superior to those of similar disperse dyes made by the methods of *BP* 420,591; 612,009, and 646,459 (J.S.D.C., **51**, 191 (1935); **65**, 187 (1949); **67**, 160 (1951)). E.T.

Colorants for Lipsticks and Nail Varnish
Thomas Kerfoot & Co. *BP* 820,111

Lakes of fluorescent dyes precipitated in presence of synthetic resins as described in *BP* 733,856 (J.S.D.C., **71**, 627 (1955)) when powdered so that they pass a 300-mesh sieve are used in lipsticks and nail varnish to obtain increased brilliance of colour in daylight. C.O.C.

Phthalocyanine Sulphur Dyes
CFM *BP* 816,656

Greenish or bluish olive-grey sulphur dyes are made by reacting a polyamino-phthalocyanine or its metal complex salt with chlorosulphonic acid and ≮ 5 mol. sulphur monochloride at 70–150°c. The products are similar to those obtained by the method of *BP* 792,018 (J.S.D.C., **74**, 495 (1958)) but are more easily made and have greater affinity and colour strength with considerably improved fastness to washing.

BP 816,657

Greenish or bluish olive-grey sulphur dyes similar to those claimed in *BP* 816,656 (above) are made by reacting polynitro-phthalocyanines with chlorosulphonic acid and ≮ 5 mol. sulphur monochloride at 70–150°c. for 2–48 hr. Reduction of the NO_2 groups to NH_2 groups and introduction of S atoms into the polyaminophthalocyanines takes place in one operation. E.T.

Water-insoluble Diazonium Compounds
Ozalid Co. *BP* 817,001

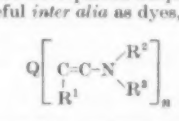
Water-soluble diazonium compounds are rendered insoluble in water by mixing them with a finely divided water-insoluble substance having groups capable of cationic exchange. Thus a solution of the zinc chloride double salt of *NN*-diethylaminobenzene-*p*-diazonium chloride (3 g.) and citric acid (3) in water (50 c.c.) are added to a dispersion of Wilkinite (3) in water (50 c.c.). The resulting mixture is filtered and the cake washed with 5% citric acid and then water to remove excess diazonium compound. The cake is dried at room temperature in a desiccator over KOH. When ground it yields a free-flowing powder containing 13% by weight of the diazonium compound. C.O.C.

Stable Concentrated Solutions of Azoic Coupling Components
American Cyanamid Co. *USP* 2,883,258

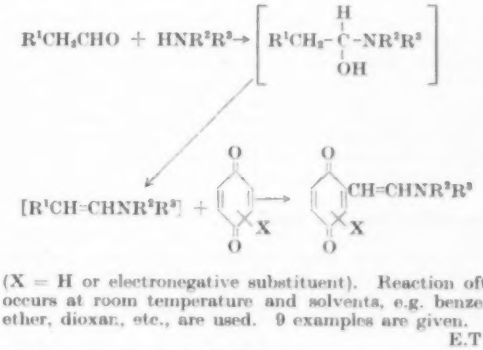
Stable 20–40% solutions of azoic coupling components are obtained in a mixture of water, ethylene glycol and if desired alcohol. The amount of ethylene glycol must not be < 5% of the total composition and the glycol and alcohol together form 20–50% of the total composition. Presence of 1% of a protective colloid prevents the composition becoming cloudy when diluted. 105 examples are given. C.O.C.

Aminovinyl Substituted Quinones
National Research Development Corpn. *BP* 819,654

Compounds useful *inter alia* as dyes, are—



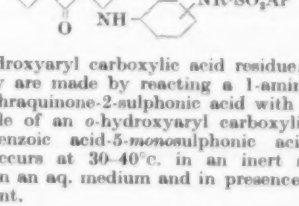
(Q = subst. or unsubst. quinone; R^1 = H, or Alk of 1–5 C; R^2 = Alk or hydroxyalk; R^3 = Alk, hydroxyalk, aralkyl, or aryl; R^2R^3 may form saturated ring with the N; n = 1 or 2). They are made by reacting quinones with aldehydes of formula R^1CH_2CHO in presence of *sec.*-amines (HNR^2R^3) thus—



(X = H or electronegative substituent). Reaction often occurs at room temperature and solvents, e.g. benzene, ether, dioxan, etc., are used. 9 examples are given. E.T.

Anthraquinone Mordant Dyes
FBy *BP* 819,956

Blue afterchrome dyes very fast to milling are—

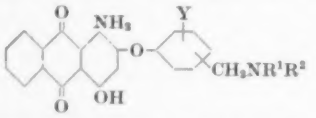


(Ar = *o*-hydroxyaryl carboxylic acid residue; R = H or Alk). They are made by reacting a 1-amino-4-(amino-anilido)-anthraquinone-2-sulphonic acid with a sulphonic acid chloride of an *o*-hydroxyaryl carboxylic acid, e.g. 2-hydroxybenzoic acid-5-*mono*sulphonic acid chloride. Reaction occurs at 30–40°c. in an inert solvent and preferably in an aq. medium and in presence of an acid-binding agent. E.T.

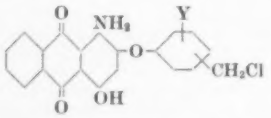
Anthraquinonyloxy-Benzyl-Amine Dyes

DuP *USP* 2,888,467

Red dyes for synthetic fibres, especially acid-modified polyester or acrylic fibres, are either quaternary ammonium or tertiary amine salts of bases of formula—



(Y = H, CH_3, OCH_3; NR^1R^2 = dialkylamino, diethanolamino, monoalkyl-monoethanolamino, monoalkyl-monobenzylamino, monoethanol-monobenzylamino, morpholino or piperidino in which the Alk group has > 4 C). They are soluble in slightly acidified hot water and, applied as basic dyes, give dyeings of good fastness to light, sublimation and washing. The intermediate—

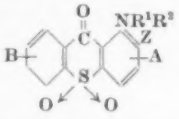


is prepared by treating a 1-amino-4-hydroxy-2-phenoxyanthraquinone in 96% H_2SO_4 at 0–10°c. with bis-chloromethyl ether. The separated and purified intermediate is then reacted with e.g. triethylamine in chlorobenzene at 95°c. and gives the red dye [*p*-(1-amino-4-hydroxy-2-anthraquinonyloxy)benzyl]triethylammonium chloride. E.T.

1-Aminobenzophenone Sulphones as Disperse Dyes

BASF *USP* 2,884,300

Very fast clear yellows to orange reds are obtained on cellulose acetate and synthetic polymer fibres by use as disperse dyes of compounds of formula—

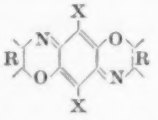


(R^1 and R^2 = same or different, H, Alk, hydroxyalkyl, cycloalkyl, aralkyl or Ar, or together form a satd. ring; Z = H, Alk, acyl, carboxylic acid or ester group; A and B = H, Alk, alkoxy, nitro or Hal). Thus 1-amino-4-methylbenzophenone sulphone yields clear yellows while 1-hydroxyethylamino-2-methyl-4-methoxybenzophenone sulphone yields orange–red dyeings. Several methods of preparing these dyes are described and 11 examples given. C.O.C.

Dioxazine Sulphur Dyes

CFM *BP* 815,840

Sulphur dyes from dioxazine derivatives brighter, faster, and stronger than those made by the methods of *USP* 2,504,153 and *GP* 717,074 are claimed. They are made by (*a*) converting a dioxazine of formula—

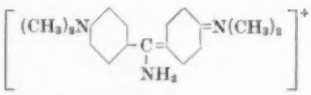


(R = iso- or hetero-cyclic residue; X = Hal) or an SO_3H derivative into the sulphochloride derivative by heating with chlorosulphonic acid, and (*b*) treating the product with an acid reducing agent, e.g. Fe and HCl. The starting materials are obtained by condensing aromatic amines and quinone (ref. *BP* 8886/1912). Advantages of the present method of manufacture are that high reaction temperatures are unnecessary and removal of large amounts of S from the products is avoided. E.T.

Alcohol-soluble Auramine Dyes

BASF *BP* 815,966

Dyes for colouring lacquers and for making plexographic printing colours are of formula $A^+ X^-$ where A^+ is, e.g.—



and X^- is, e.g. NO_3^- or NO_2^-. Generally, dyes of the auramine series are made in the form of hydrochlorides which, whilst having good water-solubility, are insufficiently soluble in alcohols for use in lacquers or printing colours. The new products are made by reacting the usual auramine hydrochlorides with, e.g. Na, K, NH_4, Ca nitrates or Na, K, Ba nitrites in water at 60–80°c. The precipitated products have excellent solubility in alcohol. E.T.

Fluorescent Brightening Agents

FBy *BP* 815,742

The products obtained by treating a water-soluble fluorescent brightening agent containing a sulphonic or carboxylic acid group with a basic condensate of HCHO and a compound containing the N–C–N group are insoluble in water. They retain the fluorescent properties of the agent from which they were made and are particularly valuable as fluorescent brightening agents for use in spinning or casting masses from which fibres, films, etc., are to be produced. C.O.C.

Stable *leuco*-Dibenzanthrone Vat Dyes

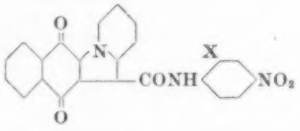
Pittsburgh Coke & Chemical Co. *USP* 2,882,280

Water-insoluble products which are stable, in presence of dil. alkali, to air and/or steam at temperatures up to 80°c. are obtained by reacting a carboxylic *leuco*-anthraquinone vat dye in water at pH 7–14 with a reducing agent and an aldehyde of < 2 C atoms. The aldehyde, e.g. glyoxal, pyruvic aldehyde, chloral, salicylaldehyde, etc., may be used in the ratio of 1 to 170 mol. of the anthraquinone vat dye. Examples of suitable vat dyes are C.I. 59825, C.I. 59800 and their halogenated derivs. The products yields prints on cotton of high tinctorial strength. When applied by pad–steam or conventional printing methods much smaller amounts of reducing agent are required than would be needed with ordinary dispersed vat dyes. E.T.

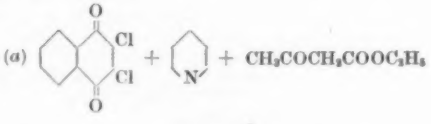
Red Pyrrocoline Pigments

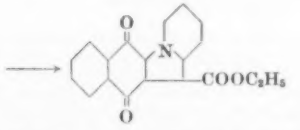
American Cyanamid *USP* 2,877,230

Red pigments possessing the brightness usually associated with azo pigments and the light-fastness usually associated with anthraquinone pigments are aromatic amides of 2,3-phthaloylpyrrocoline-1-carboxylic acids; thus—



(X = Cl, lower Alk, alkoxy or NO_2). They are also described as naphtho-(2,3-b)-pyrrocoline-6,11-dione derivs. and are made as follows—





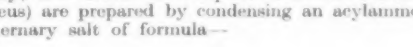
(*b*) the ester is hydrolysed to the carboxylic acid (*c*) this product is converted to the –COCl deriv. and (*d*) this carboxylic acid chloride is condensed with an appropriate nitroaniline deriv. E.T.

Cyanine Dyes

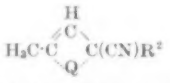
Eastman Kodak Co. *USP* 2,882,158

Dyes of formula—

(R^1 = Alk; n and d = same or different, 0 or 1; Q = atoms to complete a cyclohexane ring; R^2 = cyano or carbalkoxy; Z = atoms to complete a 5- or 6-membered nucleus) are prepared by condensing an acylammonium quaternary salt of formula—



(X = acid radical; R^3 = alkylmercapto or arylmercapto) with a compound of formula—



Thus 3-methyl-2-thiothiazolidinone and methyl *p*-toluenesulphonate were heated together and then cooled. To them was then added 3-dicyanomethylene-1,5,5,-trimethyl-1-cyclohexene, triethylamine and a little pyridine and the mixture refluxed for 10 min. On diluting with water the dye 2-[(3-dicyanomethylene-5,5-dimethyl-1-cyclohexene-1-yl)methylene]-3-methylthiazolidone was precipitated and purified by recrystallisation from alcohol. C.O.C.

Cyanine Dye Bases containing an Azaindenyl Nucleus
Eastman Kodak Co. *USP* 2,887,480

Dye bases of formula—

(R^1 = Alk; R^2 = tetrazaindenyl or pentazaindenyl; d = 0, 1 or 2; Z = atoms to complete a benzothiazole or benzoxazole nucleus), e.g. 5-[(3-methyl-2(3*H*)-benzothiazolylidene)-methyl]—tetrazolo[*a*]pyrimidine, have photosensitising properties. C.O.C.

***mero*Cyanine Dyes**
Eastman Kodak Co. *USP* 2,882,159

Dyes of formula—

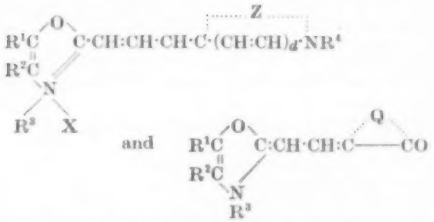
(R = Alk; d and n = same or different, 0 or 1; D = atoms to complete a cyclopentane or cyclohexane ring; Q = atoms to complete an indadione nucleus or a 5- or 6-membered heterocyclic ring), e.g. 4[2-(3-ethyl-2(3*H*)-benzothiazolyidene)cyclopentylidene]-3-methyl-1-phenyl-5-pyrazolone, have photosensitising properties. C.O.C.

Cyanine Dyes containing a 5,6-Dihydro-4-*H*-pyrano(3,2d)thiazole Nucleus
Sperry Rand Corpn. *USP* 2,886,565

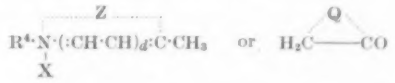
Cyanine dyes in which one or both of the auxochromic N atoms are in a 5,6-dihydro-4-*H*-pyrano(3,2d)thiazole nucleus are obtained by first converting a 2-alkyl-5,6-dihydro-4-*H*-pyrano(3,2d)thiazole into a quaternary salt by treating with an ester. This salt can then be treated in various ways to produce cyanine dyes, thus treatment with an *α*-halogenoquinoline quaternary salt in presence of an acid binding agent yields a pseudocyanine dye. If a 2-halogenopyridine quaternary salt is used then a pyridocyanine dye results. Similarly use of an ester of an ortho acid yields a symmetrical carbocyanine dye, e.g. 2-methyl-5,6-dihydro-4-*H*-pyrano(3,2d)thiazole ethiodide refluxed with diethoxymethylacetate in pyridine yields the dye 1,1′-diethylbis(5,6-dihydro-4-*H*-pyrano(3,2d)thiazolo-carbocyanine iodide whose methanol solution has a maximum absorption at 580 mμ. C.O.C.

Cyanine Dyes containing a Diaryloxazole Nucleus
Kodak *BP* 816,014

Dyes of formula—



(R^1 and R^2 = same or different Ar; R^3 = Alk, aralkyl or Ar; X = anion; d = 0 or 1; Z = atoms to complete a 5- or 6-membered ring when d = 0 or a 6-membered ring when d = 1; Q = atoms to complete a 5- or 6-membered heterocyclic nucleus; R^4 = subst. or unsubst. Alk or, when Z = atoms to complete an oxazole nucleus may = Ar) are obtained by treating an oxazole quaternary halide with an alkyl isoformanilide and then condensing the product with a compound of formula—



Thus 2-methyl-3-octyl-4,5-diphenyloxazolium iodide when heated with isoformanilide yields 2,2′-anilinovinyl-3-octyl-4,5-diphenyloxazolium iodide. This when refluxed with 3-ethyl-2-thiooxazolid-4-one in presence of acetic anhydride, triethylamine, and ethanol yields the reddish yellow dye 3-ethyl-5-(3-octyl-4,5-diphenyloxazolin-2-ylidene-ethylidene)-2-thio-oxazolid-4-one. C.O.C.

Antimony Oxide-Silica Pigment
National Lead Co. *USP* 2,882,178

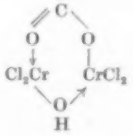
A pigment of good resistance to staining and humidity is obtained by preparing a slurry of antimony oxide, silicon and a caustic, filtering, drying and then calcining. The product is suitable and economical for use in both flame-retardant and exterior paints. C.O.C.

Pigments containing Oxygen and Silicon for Reinforcing Rubber
B. F. Goodrich Co. *USP* 2,882,177

Inorganic pigments containing O and Si, e.g. Silica (C.I. Pigment White 27), silicon monoxide [$Si_x(SiO_2)_y$] and silicon oxyimide, when treated with an organic polyhydroxy compound, e.g. glycerylmonolaurate, have reinforcing properties equal to those of Carbon Black (C.I. Pigment Black 6 and 7). C.O.C.

Siliceous Pigments
Columbia–Southern Chemical Corpn. *USP* 2,884,402

Finely divided hydrated silica of composition $H_2O\cdot(SiO_2)_{3-85}$ when treated with an organo-metallic complex of formula—



(R = residue of a carboxylic acid) e.g. stearato chromic chloride, is an excellent reinforcing pigment for use in natural or synthetic rubber. C.O.C.

Hydrophobic Silica (C.I. Pigment White 27)
General Electric Co. *BP* 816,337

Silica is rendered hydrophobic by treating it with an alcohol in presence of a hydrohalic acid at 65–150°c. C.O.C.

Silica (C.I. Pigment White 6)
Columbia–Southern Chemical Corpn. *BP* 816,686

Finely-divided precipitated silica useful as a rubber or paper pigment is obtained by mixing in any sequence

finely-divided silica, a source of alkaline earth metal ions and a source of sulphite ions. Thus SO_2 was bubbled through aqueous sodium silicate, when the stoichiometric amount of SO_2 had been used its entry was stopped and the resulting slurry was boiled for 1 hr. 30% excess $CaCl_2$ (based on the equivalent Na_2O content) was then added followed by adding milk of lime to bring the mixture to pH 7·1. The pigment was then washed and dried. C.O.C.

Titanium Dioxide (C.I. Pigment White 6)
Laporte Titanium *BP* 815,891

Titanium tetrahalide is oxidised by air in a fluidised bed at 750–1250°c. The $TiCl_4$ vapour contains $TiCl_3$ as rutile promoter or $AlCl_3$ or $SiCl_4$ as a conditioning agent. The TiO_2 formed is carried away by the gases leaving the fluidised bed. C.O.C.

Yield Seed for Preparing Titanium Dioxide (C.I. Pigment White 6)
National Lead Co. *USP* 2,886,415

Method of preparing yield seed of unusually high potency in the hydrolysing of titanium salt solutions. C.O.C.

Vinylic Filler Pigments
O. W. Burke *BP* 820,207

A vinylic filler, i.e. the insoluble vinyl polymers described in *BP* 799,043, is used in combination with an organic and/or inorganic colorant by treating the filler with an aqueous suspension or solution of the colorant. Their properties and uses vary widely depending upon the colorant used. 80 examples are given covering the use of every class of dye or pigment as the colorant. C.O.C.

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7) (I p. 546)
Lead Titanate Paints in Holland and Lead Titanate as a Pigment (V below)
Colouring Cellulosic Textiles with Solubilised Phthalocyanine Dyes (VIII p. 570)
Elucidation of the Mechanism of Colour Development by Investigation of the Kinetics of the Reaction (IX p. 571)

V— PAINTS; ENAMELS; INKS

Effect of Raw Materials on the Leafing of Aluminium (C.I. Pigment Metal 1) in Varnishes
H. Pfau and A. Grossmann
Farbe u. Lack, **65**, 179–184 (1959)

Effects of solvents and resins on the leafing properties of aluminium flake were investigated at 15% binder and 12% pigment content by the spatula (ASTM D–480–51) test. Generally, initial leafing increased with increasing d of the solvent. Aromatics, Decalin, cyclohexanol, diacetone alcohol, and esters impart best retention of leafing. High acid number decreases the stability of leafing in rosin, coumarone and alkyd vehicles but not in phenolics. Viscosity and chemical composition have only a minor effect. C.O.C.

Surface Coatings derived from Tall Oil. I— Aluminium Complexes may provide Complete Paint Vehicles. II, III, and IV
J. Weiss *Paint Manuf.*, **28**, 112–116, 169–172, 243–246, 303–306, 312 (1958):
Chem. Abs., **53**, 9688 (25 May 1959)

Work showing that Al complexes based on tall oil are suitable drying oils for use in paints and varnishes. C.O.C.

Lead Titanate Paints in Holland and Lead Titanate as a Pigment
J. Rinse
Paint Varnish Production, **48**, (13), 98–100 (1958):
Chem. Abs., **53**, 9687 (25 May 1959)

$PbTiO_3$ can be made by calcining TiO_2 (27 parts) and PbO (73) at 600–800°c. in presence of a catalyst. The superior weathering properties of $PbTiO_3$–alkyd paints have led to their general use in Holland. The differences in application and performance requirements between Dutch and U.S.A. house paints are pointed out. The properties of $PbTiO_3$ as a pigment for various types of paint are considered. C.O.C.

Effect of Temperature on Sedimentation Volume of Pigment Suspensions
L. Dintenfass *Kolloid-Z.*, **162**, 47 (1959):
Chem. Abs., **53**, 8658 (10 May 1959)

In concentrated pigment dispersions the sedimentation volume F increases with increase in viscosity of the liquid phase. F of deflocculated pigment suspensions is unaffected by change in temperature. F of flocculated pigment suspensions increases with increase in temperature. This can be reversed quantitatively by simultaneously lowering the temperature and tapping the glass tube. C.O.C.

Thixotropy of Concentrated Pigment Dispersions
L. Dintenfass *Kolloid-Z.*, **163**, 48–68 (1959):
Chem. Abs., **53**, 11947 (10 July 1959)

The thixotropy and viscosity of concentrated pigment suspensions in Newtonian liquids, e.g. toluene, paraffin, glycerol, etc., were investigated with a rotational viscometer. The sedimentation volume of pigment suspensions and the degree of flocculation of the pigment are the main parameters; the others are liquid viscosity, temperature and the viscosity of suspension above the critical rate of shear. The thixotropic effect is related to the "slope" of the rheological curve plotted on the log–log graph as kinematic viscosity against the rate of shear. The slope increases with increase in sedimentation volume, F, and with increase in the degree of flocculation, β, which is measured as the ratio of the sedimentation volume of the sample to that of a suspension of the same pigment at absolute pigment dispersion. Increase of pigment concentration caused a marked "crowding effect" that increased expotentially. The slope, S, is influenced by this effect and by a "compression effect" when the pigment-free volume is > 1. Increase in the viscosity of the liquids increased the sedimentation volume, more so at higher values of β; the slope of the rheological plot remains constant if pigment concentration and degree of flocculation are both constant. Change of temperature changes sedimentation volume and slope. Increase of temperature, therefore, increases the thixotropy of suspensions, as well as reduces the viscosity of the liquid. Increased rate of shear is associated with shearing-off lyospheres and the mechanically induced nonequilibrilised dispersion of pigment particles. Plotting viscosity against rate of shear on log–log paper showed the fallacy of "yield value" as an intercept of stress-axis and the extrapolated rheological curve, drawn on a graph. The yield value as such is a time dependent function. Only the equilibrium states are discussed and the inter-relations of various parameters, with some semiquantitative equations. C.O.C.

Ultraviolet-absorptive Additives in Lacquers and Plastics
H. Stecher *Adhäsion*, **2**, (6), 243–244 (1958):
Chem. Abs., **53**, 14579 (10 July 1959)

The following compounds are efficient ultraviolet absorptives (upper limit of their absorption range using 3 g./sq.m. is given): flavone, 7-hydroxy 354 mμ., 5,7-dihydroxy 397 mμ., 5,7,4′-trihydroxy 401 mμ., 3,5,7,4′-tetrahydroxy 413 mμ., 3,5,7,3′,4′-pentahydroxy 422 mμ., 3,5,7,3′,4′,5′-hexahydroxy 427 mμ.; benzalacetophenone 360 mμ.; 2,4,3′,4′-tetrahydroxybenzalacetophenone 470 mμ.; benzophenones, 2-hydroxy-4-methoxy 350 mμ., 2,2′-dihydroxy-4-methoxy 380 mμ., 2,2′-dihydroxy-4,4′-dimethoxy 400 mμ. C.O.C.

Removal of Sulphide Stains from Paints containing Lead
E. Hoffman
Paint J. Australia New Zealand, **3**, (9), 9–10 (1958):
Chem. Abs., **53**, 11855 (25 June 1959)

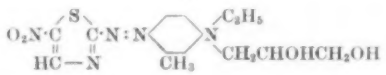
Stains caused by action of H_2S on paints containing lead may be removed with 10-vol. H_2O_2. A more permanent removal is obtained by use of 1 oz. disodium ethylene diamine tetraacetic acid in 0·75 pint of 10-vol. H_2O_2. The solution is applied, allowed to dry, washed with water, and the process repeated until the stain is removed. C.O.C.

PATENTS

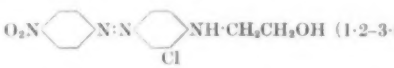
Ink for Marking Plastic Materials

Eastman Kodak Co. *USP* 2,879,168

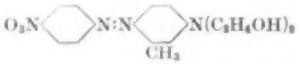
An ink which is applicable to both hydrophobic and hydrophilic surfaces contains 2-furaldehyde (generally > 50% of the ink), an organic solid, e.g. powdered or flaked cellulose ester (0·5–5·0%) and a solvent. The colorant is preferably a mixture of at least two organic dyes. Thus an ink which is quickly absorbed by high acetyl cellulose acetate film to yield a black mark consists of 2-furaldehyde (60–97%), powdered cellulose acetate butyrate (1·2–3·0), C.I. Solvent Yellow 2 (10), the blue dye—



(1·2–3·0) and the orange dye—



If a red mark is required then the above mixture of dyes is replaced by the red dye—



C.O.C.

Chromic Hydroxide— Ferric Hydroxide Compound for Increasing the Durability of Varnish Films

DuP *USP* 2,879,246

Addition to varnish or other coating composition of 2–50 parts per 100 of film-forming material of a "pigment" comprising 70–95 mol.% chromic hydroxide and 5–30 mol.% ferric hydroxide much lengthens the life of films formed from the coating. The addition of this "pigment" neither colours nor renders the film opaque and has no effect on colorants added to the composition. C.O.C.

Paint Vehicle having Fungicidal Properties

U.S. Secretary of Agriculture *USP* 2,884,330

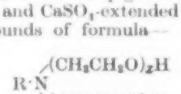
The drying oil in a paint vehicle has tripropionin or undecylenic acid incorporated in it by ester exchange, e.g. by the tripropionin being added before the vehicle is heat bodied, or hexachlorocyclopentadiene by Diels–Alder addition. The whole of the product is then resistant to fungi and migration of the fungicide is impossible.

C.O.C.

Stabilised Latex Coating Compositions containing Reactive Pigments and Glycolamine Stabilisers

Dow Chemical Co. *USP* 2,887,460

Aqueous colloidal suspensions containing film-forming organic polymers, reactive pigments, e.g. ZnO (C.I. Pigment White 4) and $CaSO_4$-extended TiO_2 are stabilised by use of compounds of formula—



(R = aliphatic radical of > 7 C; $x + y = \leq 2$). The stabilised dispersions are used in paints, etc. C.O.C.

Preserving the Gloss in Paint Films during Drying

Sherwin-Williams Co. *USP* 2,886,456

Addition of 0·5–2·0% of a sulpho-succinate, e.g. dioctyl-, di-(2-ethylhexyl)- or di-(3,7-dimethyloctyl)succinate, prevents freshly applied paint films from being flatted by atmospheric moisture. C.O.C.

Silicone Coatings for producing a Hammer Finish

Dow Corning Corpn. *USP* 2,884,388

A hammer finish can be produced with all known types of coating resins if a benzene-soluble organopolysiloxane of viscosity ≤ 50,000 cs. at 25°c. is incorporated in the coating composition. 0·01–2·0% of the siloxane on the wt. of resin is needed. C.O.C.

Coating Compositions containing Metal Particles

American Marietta Co. *BP* 815,793

A translucent film-forming medium contains plate-like particles of metal 3–15 millionths of an inch thick and having reflection coefficient ≤ 35% and light transmission coefficient ≤ 5%. Good results are obtained by presence of 0·02–0·2% by wt. of the metal, especially Al and Cr. It is preferable that the metal particles can pass through a 400 meshes per inch sieve (U.S. Standard). C.O.C.

Powdered Paint Readily Miscible with Water

United Gilsonite Laboratories *USP* 2,880,104

A stable water-reducible powdered texture paint consists of a protein, a water-soluble cellulose ether, Talc (C.I. Pigment White 26) or Mica (C.I. Pigment White 20) as embodying agent, a filler, pigment, a solubiliser, and a fungicide, e.g. Mg silicate (68–77 lb.), hydrated aluminium silicate (66–74), mica (53–60), $CaCO_3$ (C.I. Pigment White 18) (30), titanium calcium pigment (15–20), water-soluble cellulose ether (55% active cellulose) 5–10, Na pentachlorophenate (1–3), borax (1–3), and protein (30–40).

C.O.C.

Aqueous Emulsion Paints containing Cyanoethylated Starch

Monsanto Chemical Co. *USP* 2,881,143

Use of cyanoethylated starch as the stabiliser or dispersing agent in aqueous emulsion paints yields a paint resistant to attack by micro organisms. C.O.C.

Surfactants derived from Acrylates and Methacrylates (III p. 547)
Solutions of Nylon as Coating Compositions for Textiles, Paper, Metals, etc. (III p. 549)
Antimony Oxide–Silica Pigment (IV p. 563)
Rendering Polyethylene Surfaces Adherent to Hydrolysed Vinyl Chloride–Vinyl Acetate Copolymers, Polyvinylalkyl Ethers, and Acrylic or Methacrylic Polyesters (XIII p. 577)

VI— FIBRES; YARNS; FABRICS

X-Ray Diffraction Patterns of Cotton at and between Reversals

H. Wakeham, T. Radhakrishnan, and G. S. Viswanathan
Text. Research J., **29**, 450 (May 1959)

Evidence is presented that at points of structural reversal in cotton fibres there is a high degree of longitudinal order. The X-ray microcamera technique employed did not permit quantitative evaluation of fibre crystallinity to determine whether the reversals were also of higher crystallinity than the rest of the fibre.

S.B.D.

Motion of Water Molecules on Fibrous Materials— I

A. Odajima, S. Inoue, and J. Hayashi
Hokkaidô Daigaku Ôyô Denki Kenkyûjo Ihô, **10**, 95–101 (1958):
Chem. Abs., **53**, 11998 (10 July 1959)

The proton magnetic resonance absorption of water adsorbed on native cellulose was measured at water content, W, of 10–180% at 20°c., using a Varian spectrometer. With $W > 10\%$ no absorption was seen. Sharp narrowing was found at about $W = 20\%$. The correlation time derived from the half width of absorption by assuming $T_2 = 5 \times 10^{-6}$ sec. was greater than the relaxation time derived from the dielectric absorption by a factor of 10. This is accounted for by assuming anisotropy in the ease with which the adsorbed water molecules rotate. The line-width transition indicates that the absorbed water exists as two types of mobile water molecules.

C.O.C.

Action of Light on Cotton Cellulose. I— Chemical Modification of the Macromolecule of Cotton caused by Artificial Light

F. I. Sadov and R. I. Vylcheva
Izvest. Vysshikh Ucheb. Zavendii, Tekhnol. Tekstil. Prom., (6), 66–75 (1958):
Chem. Abs., **53**, 12687 (10 July 1959)

Artificial light causes oxidation of primary and secondary hydroxyl groups and formation not only of aldehyde and carboxyl groups but also keto groups. Cotton bleached with hypochlorite is degraded more by exposure to artificial light than cotton bleached by other agents.

C.O.C.

Influence of High-energy Radiation on Cotton

I—Properties of Raw Cotton Irradiated Under Various Conditions

H.-P. Pan, B. E. Proctor, S. A. Goldblith, H. M. Morgan, and R. Z. Naar

Text. Research J., **29**, 415–421 (May 1959)

Raw cotton of known composition was irradiated with cathode rays under various conditions and in various forms. Some yarns were also irradiated with gamma rays. Degradation was found to have taken place in the irradiated cottons and their direct dye affinity was similar to that of oxycellulose. A new absorption peak was found in the infrared spectra of cotton irradiated at high dose levels. The tensile strength of yarns decreased with increase in dosage, but at low dose levels the tensile strength was found to increase to a maximum before it started to decrease. A general increase in mildew resistance was noted in irradiated samples compared with unirradiated samples.

II—Effects of Chemical Modification

Idem *Ibid.*, 422–425

Mercerised cotton and partially acetylated cotton yarns were irradiated with cathode rays. Mercerised yarns and partially acetylated yarns were also prepared from previously irradiated cotton yarn. The behaviour patterns of the properties of these samples were found to be similar to those of the raw cotton yarn from which they were prepared.

III—Test for Degradation

H.-P. Pan, B. E. Proctor, and S. A. Goldblith

Ibid., 425–430

Raw cottons irradiated with cathode rays were found to form coloured compounds with 2-thiobarbituric acid in acidic soln. A reproducible, rapid, and direct test method was evolved, the results comparing well with those from other data. Microbiologically deteriorated cottons were also found to be sensitive to this test. The possibility of using this test for the rapid determination of chemical damage to raw cotton and other substances that contain cellulose as the major constituent is suggested. S.B.D.

Breaking Strength of Hydrolytically Degraded Yarns, Mercerised and Unmercerised

S. S. Trivedi and A. G. Chitale

J. Textile Inst., **50**, T 390–T 392 (May 1959)

Mercerisation makes cotton more susceptible to chemical attack, but nevertheless mercerised specimens show higher strength than unmercerised ones when degraded by hypochlorite oxidation, ultraviolet radiation, and sunlight and weathering. The present results show that this generalisation can be extended to include hydrolytic degradation by N hydrochloric acid for several days at room temperature. P.T.S.

Crimp in Alkali-treated Jute Fibres

M. Lewin, M. Shiloh, and J. Banbaji

Text. Research J., **29**, 373–385 (May 1959)

The crimp diameter, crimp ratio, extension, and wave number are quantitatively determined for various jute samples under varying conditions. No crimp is formed with less than 6% NaOH, the parameters rising to maximum values at 9% NaOH, then decreasing to constant values with increased amounts of alkali. Crimp is formed within 30 min. The optimum temperature for crimping is 2°c. With increase in temperatures the values of the crimp parameters decrease until at 40°c. no crimp is observed. S.B.D.

Movement of Wool Fibres during Felting

K. R. Makinson

Text. Research J., **29**, 431–439 (May 1959)

I—Light Felting in a Pleated Worsted Fabric

Pleats in worsted fabric which were fast to relaxation in hot water were readily destroyed by light hand washing. This was shown to be due to small movements of the fibres, which were the initial stage of felting. The pleats were destroyed by blurring the position of the region of maximum set and by imposition of temporary opposing sets on the fibres.

II—Heavier Felting in a Worsted Fabric

Ibid., 439–443

Before there was any perceptible felting whilst washing worsted fabric, small movements of portions of some fibres towards their root ends occurred. Further washing caused loops to form in some parts of the fibres while other parts were taut, and felting and shrinkage became apparent. At a later stage of felting entanglement of the loops occurred. The agreement of this behaviour with various theories of felting was discussed. S.B.D.

Stress-Relaxation in Keratin Fibres

M. Feughelman and T. W. Mitchell

Text. Research J., **29**, 404–409 (May 1959)

Variation of force with time in water for single wool and hair fibres stretched to both 10% and 40% extension was measured at various temperatures between 20°c. and 100°c. It was shown that at 100°c. and 40% extension the force after 1 hr. was mainly elastomeric, and that between 60° and 70°c. a second order transition occurs for the stretched wool fibre in water. Chemical treatments which accelerate or retard the ability of a fibre to permanently set also reduce or increase value of the force after 1 hr. S.B.D.

Discoloration of Wool Fibres by a Fungus

A. P. Mulcock *Nature*, **183**, 1281–1282 (2 May 1959)

A fungus of the species *Peyronellaca* causes black discoloration of stored wool. The colour is stable to aqueous scouring and solvent extraction. Although the stained fibres appear to be intact, they have a very low tensile strength, and under the microscope the fungal hyphae are easily seen. E.V.T.

Action of Copper Ammines on Wool

C. S. Whewell, J. Ashworth, V. R. Srinivassan, and A. G. P. Vassiliadis

Text. Research J., **29**, 386–393 (May 1959)

When wool is immersed in soln. of metal ammines, the metal is adsorbed and the fibres contract. There are also changes in the X-ray photographs of the fibres. Data are given for the absorption of copper from several copper ammines by untreated and chemically modified wools. The mechanism of the reactions involved is discussed. The effect of the absorbed copper on the physical properties of the fibres is also considered, special attention being given to the moisture relations and the setting characteristics of the metal-containing fibres. S.B.D.

Shrinkage of 6,6-Nylon Yarns in Steam

D. L. Munden and D. Slater

J. Textile Inst., **50**, T 393–T 403 (June 1959)

The experiments described here show that the free shrinkage of 6,6-nylon yarn in steam depends on the previous thermal treatment of the yarn. It can thus be used to investigate such treatments. Industrial examples are given, and it is suggested that similar results may be obtained with other thermoplastic fibres, e.g. Terylene, Perlon. P.T.S.

Effect of Heat on Unoriented Amorphous Polyethylene Terephthalate Fibres

S. Sakajiri

J. Soc. Textile Cellulose Ind. Japan, **15**, 3–5 (Jan. 1959)

Polyethylene terephthalate fibres of different degrees of polymerisation and orientation are examined by following the changes in sp.gr., birefringence, and X-ray diffraction patterns before and after heat treatment. The onset of crystallisation was shown by the sp.gr. changing from 1·339 to 1·384 (slack) and to 1·382 (under tension) when $|\eta| = 0{\cdot}51$ and from 1·343 to 1·382 (slack) and 1·381 (under tension) when $|\eta| = 0{\cdot}62$. The birefringence also changes from about $+0{\cdot}007$ to $-0{\cdot}002$ or $-0{\cdot}009$. Similar conclusions are drawn from the X-ray intensity measurements. L.P.

Effect of Heat and Chemicals on Diolen Polyester Fibre

W. Brennecke and H. Richter

Melliand Textilber., **40**, 531–536 (May 1959)

The authors tabulate and illustrate graphically the effect of fifty chemicals, including organic and inorganic acids, alkalis, oxidising and reducing agents, organic solvents, and metallic salts, on Diolen, at 40, 70, and 95°c., for times up to 72 hr., emphasising changes in weight and in tenacity. Heat stability up to 220°c. is evaluated. A useful compendium. Eight references dealing with similar investigations are cited. Swelling with formic and acetic acid, surface hydrolysis by alkali, loss of tenacity with ammonia despite weight constancy, and shrinkage in dichloromethane and chloroform are singled out for comment. S.M.J.

Effect of Organic Solvents on Polyethylene Terephthalate Fibres
S. Sakajira
J. Soc. Textile Cellulose Ind. Japan, **15**, 6–8 (Jan. 1959)
The changes in birefringence, sp.gr., and X-ray diffraction pattern of polyethylene terephthalate fibres brought about by their immersion in water, methanol, ethanol, glycerin, benzene, xylol, benzyl alcohol, acetone, dioxane or nitric acid are reported. There was generally a decrease in birefringence except after immersion in acetone or nitric acid. The sp.gr. usually increased and increase in crystallisation was confirmed by the X-ray intensities. The shrinkages brought about by immersion varied with the degree of polymerisation but not with birefringence. L.P.

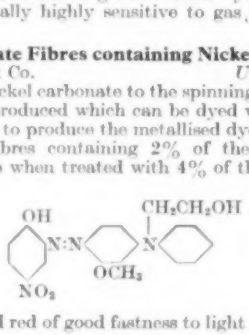
PATENTS

Cellulose-alkali-biuret-copper (or Nickel) Complexes
L. L. Balassa *USP* 2,887,398
Cellulose is dissolved in an alkali-biuret-copper (or nickel) solution and then extended into a coagulating bath to produce filaments or film. The products contain a copper or nickel compound intimately bound or entrained in the cellulose structure and as a result have good rot-proof properties. Presence of the metal salt in the fibre also enables intimate bonding of addition compounds with the cellulose, e.g. with dyes capable of forming a lake with copper or nickel. C.O.C.

Reducing the Water Imbibition of Regenerated Cellulose Threads
Courtaulds *BP* 817,294
Treatment with a compound containing at least two 2,3-epoxypropyl groups followed by heating is used. Thus viscose staple fibres having a water imbibition of 98% were passed through an aqueous solution of tris-(2,3-epoxy propyl)amine, dried at 70°c. for 2 hr. and baked at 140°c. for 15 min. The treated fibres had a water imbibition of 48·6%. C.O.C.

Cellulose Derivative and Polyester Fibres which when Dyed had Innate Resistance to Gas Fume Fading
Celanese Corpn. of America *USP* 2,880,051
Incorporation into the fibre substance, before the fibre is formed, of an alkaline salt of a copolymer of an alkyl vinyl ether and an ethylene carboxylic acid, e.g. a copolymer of equimolecular parts of methyl vinyl ether and maleic anhydride, results in fibres which have excellent fastness to gas fume fading even when dyed with dyes which are normally highly sensitive to gas fume fading. C.O.C.

Cellulose Acetate Fibres containing Nickel Carbonate
Eastman Kodak Co. *USP* 2,882,176
Addition of nickel carbonate to the spinning dope results in fibres being produced which can be dyed with a metallisable dye so as to produce the metallised dye in the fibre. Thus acetate fibres containing 2% of their weight of nickel carbonate when treated with 4% of the dye—



at 80°c. are dyed red of good fastness to light and washing. C.O.C.

Flame-resistant Acrylonitrile Polymers
Monsanto Chemical Co. *USP* 2,881,147
Acrylonitrile polymers are rendered flame resistant by incorporation of 2-cyanoethyltetramethyldiamido phosphate $[(CH_3)_2N]_2{\cdot}PO{\cdot}OCH_2CH_2CN$. C.O.C.

Enhancing the Affinity of Vinylidene Cyanide Copolymer Fibres for Acid Dyes
B. F. Goodrich Co. *BP* 816,869
Treatment with a dilute aqueous solution of a primary or secondary amine or a secondary heterocyclic amine at a temperature which allows the amine to react with the cyano groups in the fabrics used. Thus vinylidene cyanide-vinylacetate copolymer yarn was treated for 2 hr. at 100°c. in 4·4% aqueous ethylene diamine, treated for 15 min. in a boiling solution of Na *N*-methyloleoyl taurate, dried 1 hr. at 110°c. and then dyed in a bath containing (on wt. of fibre) 5% Fast Light Red 4BA (C.I. Acid Red 34) and 6% formic acid, liquor ratio 40 : 1. The exhaustion of the dyebath after 1 hr. at 95°c. was 1·25 g. dye/l. With an untreated yarn dyed in an identical bath the exhaustion was only 0·036 g./l. C.O.C.

Modifying the Structure and Properties of Addition Polymers
DuP *BP* 820,120
The polymer is subjected to ionising radiation while in intimate contact with an organic compound, which when chemically bound to the polymer as a result of the irradiation causes appreciable change in one or more of the polymer's properties. Thus polyacrylonitrile continuous filament yarn is immersed in a 16% aq. soln. of polyethylene glycol of mol.wt. 20,000. Excess solution is squeezed from the yarn which is then wrapped in aluminium foil and subjected to electron irradiation supplied by a 1 million electron volt resonant transformer with a beam-out current of 560 micro-amps. It passes to and fro across the beam at 16 in. per min. until it has received a total dose of 40 Mrep. It is then given a hot wash with aqueous detergent, rinsed in distilled water and dried. This gives an excellent antistatic finish of good fastness to washing. C.O.C.

Polychlorotrifluoroethylene Filaments
Union Carbide Corpn. *BP* 816,987
Describes production of polychlorotrifluoroethylene filaments suitable for textile usage. They have excellent resistance to chemicals, heat, and abrasion and are very tough. C.O.C.

Dextran Solutions as Finishes for Paper and Textiles and for Making into Fibres (III p. 548)
Decreasing the Hydroxyl Number of Polyesters (XIII p. 577)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Mechanism of Detergence
A. S. C. Lawrence
Nature, **183**, 1491–1494 (30 May 1959)
On the basis of a study of the properties of ternary surface-active agent/water/amphiphile systems, an account is presented of a mechanism of detergence in terms of cryoscopic forces. The mechanism postulated suggests that detergence is not brought about by surface energy phenomena, but solely by cryoscopic forces. W.K.R.

Trichloroethylene in Continuous Scouring and Bleaching
W. A. S. White, H. J. Ross, and N. F. Crowder
J. Textile Inst., **50**, P 274–P 293 (June 1959)
Previous work on solvent extraction is reviewed. Laboratory work and plant trials show that continuous dewaxing of cotton with trichloroethylene facilitates desizing and bleaching, and is very suitable for continuous processes because the product has a high absorbency. The practical advantages of the process are stressed, and are said to be particularly useful for cotton blended with rayon, jute or Terylene, which will not stand severe alkaline treatment. The pilot plant is comprehensively described. P.T.S.

Peroxide Bleaching. II— Last-stage Bleaching of Kraft Pulp with Peroxide
E. Lindahl, C.-G. Moberg, and L. Stockman
Svensk. Papperstidning, **62**, 308–317 (15 May 1959)
A kraft pulp prebleached to 80% G.E. can be further bleached to 91% with 0·9% H_2O_2 (100%) calculated on the weight of the pulp and to 93% G.E. with 1·2% H_2O_2. The increased brightness was, however, accompanied by an undesirable decrease in viscosity. Brightness was increased by an additional 2 units on extending the time from 4 to 10 hr. at 70°c. High pulp concentrations and high temps. favour brightness. The yellowing tendency is lower than for chlorine dioxide bleached pulp. (16 references.) R.A.

Degradation of Alkali Cellulose by Hydrogen Peroxide in Caustic Soda
T. N. Kleinert *Textil-Rund.*, **14**, 249–259 (May 1959)

Peroxide decomposition is accelerated by the presence of hemicelluloses. In ripening alkali cellulose in caustic soda containing hydrogen peroxide the quantity of the latter, the temperature and the recirculation of the liquor must be adapted to the type of cellulose in order to effect an improvement in alpha cellulose content. The time should not exceed 2 hr. if small quantities of hemicellulose are present. S.R.C.

Continuous Bleaching of Cotton Fabrics
L. A. Gotovtseva, M. G. Shikher, and A. V. Surovaya
Tekstil. prom., **19**, 45–50 (May 1959)

Singed and desized grey cotton is scoured and bleached in rope form in the following sequence: (1) impregnate in hot NaOH using two mangles; (2) steam at 100°c. (55–75 min.); (3) wash in hot and cold water; (4) acidify; (5) wash in cold water; (6) impregnate with cold alkaline H_2O_2; (7) steam (55–75 min.); (8) wash off with hot and cold water. Processing details for each operation are given, and the equipment used is referred to the Russian coding system. The amount of steam, temp., acid, and alkali in H_2O_2 soln. are controlled automatically, whilst the alkali for impregnating, H_2O_2 and sodium silicate are regulated manually. Difficulties have been experienced with (*a*) cloth deformation in step 1 with satin and gauze fabrics; (*b*) high liquor uptakes throughout; (*c*) uneven scouring of some fabrics, e.g. voiles, in step 1–2 requiring a second steaming apparatus. Analysis of processed fabrics by determinations of capillarity, fluidity, whiteness, loss in weight is given, and superior results to normal pressure kier boiling and bleaching are claimed. G.J.K.

Reductive Bleaching of Mechanical Pulp with Sodium Borohydride
W. C. Mayer and C. P. Donofrio
Pulp Paper Mag. Can., **59**, (10), 157–163, 166 (1958):
Chem. Abs., **53**, 9662 (25 May 1959)

As a bleaching agent $NaBH_4$ was most effective at 35°c. 1% $NaBH_4$ on the weight of pulp gave an 8-point increase in brightness but only about one quarter of the $NaBH_4$ was actually used in bleaching, the other three-quarters being lost by hydrolysis. High pH improves the stability of $NaBH_4$ but a long bleach is necessary to obtain satisfactory brightness. At low pH the reductive and decomposition rates are more rapid. pH about 10 gives optimum results taking into consideration both loss by decomposition and bleaching time. Spectrophotometric study of liquor treated with $NaBH_4$ showed the specificity of $NaBH_4$ for the reduction of :CO groups. C.O.C.

PATENTS

Bleaching of Linen
Bleachers' Assocn. *BP* 819,637

Sodium chlorite alone adequately bleaches linen if it is applied at > 60°c. and pH < 7 from an aqueous solution containing > 10 g./l. of the chlorite. If the linen is given two such treatments and if either of these is preceded by treatment with boiling alkali then a full white is obtained which is as stable as that produced by lime boiling and hypochlorite treatment. To get a stable white it is necessary that any degradation products formed shall be removed so if a hot alkali treatment is not given then any chlorite treatment should be followed by soaping at the boil. C.O.C.

Bleaching Chemical Wood Pulp with Nitrogen Trichloride
Champion Paper & Fibre Co. *USP* 2,882,120

Brightness is much increased when chemical wood pulp is treated with nitrogen chloride in presence of water at pH $< 4{\cdot}5$. There is much less degradation of the cellulose than in ordinary methods of bleaching. The nitrogen chloride is readily formed by adding an ammonium compound to an aqueous solution of chlorine at pH 4·5. Thus semi-bleached southern pine sulphate pulp (30 g.) of brightness 73·9 and viscosity 8·3 cp. suspended in water was treated with a mixture of 10% aq. $(NH_4)_2SO_4$ (1·87 c.c.) and chlorine water (76 c.c. containing 0·3 g. available Cl). Enough water was then added to produce a pulp suspension of 10% consistency. The pH was 2·1. The slurry was gradually heated to 120°F. and 2 hr. after the NCl_3 was added the pulp was washed with water. The treated pulp had brightness 82·4 and viscosity 7·9 cp. Pulp similarly treated in absence of NCl_3 but at pH 2·6 had brightness 82·3 and viscosity 4·9 cp. C.O.C.

Bleaching Synthetic Polymer Fibres with Permonosulphuric Acid
Stevensons (Dyers) *USP* 2,882,121

Synthetic linear polymer fibres are readily bleached by aqueous solutions of permonosulphuric acid. C.O.C.

History of Dyeing and Bleaching— the Thirteen Key Books (VIII below)

VIII— DYEING

History of Dyeing and Bleaching— the Thirteen Key Books
S. M. Edelstein
Amer. Dyestuff Rep., **48**, 35–40 (4 May 1959)

The bibliograph of bleaching, dyeing, and finishing is discussed. Many of the books published before 1856 were retranslations or digestions of thirteen important books which contain the main story of the development of dyeing and bleaching from the earliest times to the middle of the nineteenth century. A bibliographical description of these thirteen key books, which were published before 1856, is given. W.P.M.

Kinetics of Dyeing
P. V. Morygenov and B. V. Mel'nikov
Izvest. Vysshikh Ucheb. Zavedenii, Tekhnol. Tekstil. Prom., (5), 96–108 (1958):
Chem. Abs., **53**, 8634 (10 May 1959)

Review, 38 references. C.O.C.

Relation between Molecular Weight and Diffusion Coefficient in Dyeing
K. Odajima J.S.D.C., **75**, 255 (May 1959)

Isoequilibrium Temperature in Dyeing
K. Odajima J.S.D.C., **75**, 308 (June 1959)

Rubbing Fastness of Azoic Dyeings
H. Keil *Textil Praxis*, **14**, 520–524 (May 1959)

After critical review of some of the factors which can affect successful dyeing the author concludes that out of well over 2,000 possible azoic combinations in the vast number of cases the fastness to rubbing is satisfactory if well established procedures are followed and necessary precautions taken. The cleansing pretreatments (scouring, bleaching, etc.) are important as well as correct choice of suitable azoic coupling components for a particular type of material and a specific purpose. Indiscriminate choice of auxiliary chemicals or finishing agents may lead to lowering of the rubbing fastness. L.A.T.

Metachromasy of Polyvinylpyrrolidone
I. Tsubomura, J. Yoshioka, and K. Torii
Bunko Kenkyu, **6**, (3), 10–14 (1957):
Chem. Abs., **53**, 8107 (10 May 1959)

Addition of polyvinylpyrrolidone (I) influences the pH at which several dyes change colour and fluorescence. With Congo Red (C.I. Direct Red 28) (II) this change is 2 pH units. Absorption spectra of II, the "halfstructure" compound of II and Methyl Orange (C.I. Acid Orange 52) (III) with and without I were measured at pH 7·0. II showed increased absorption in presence of I. The "half-structure" compound showed no increase in absorption, only shift of the wavelength of maximum absorption. III was hardly affected by presence of I. Absorption measurements carried out at various concentrations of I with constant dye concentration and at the same wavelength, showed that I added near to the saturation point caused striking increase of absorption, with the "half-structure" and III there was no increase. Combination of II and I seems to be by a H bond between the end amino group of II and the carbonyl group of I. With III H bond formation is improbable because of the CH_3 groups in the dimethylamino group. C.O.C.

Sulphur Dyes. XXXVII— Dyeing Mechanism of Pyrogene Indigo
H. Hiyama and M. Morimoto
Kagaku to Kōgyō (Osaka), **33**, 17–21 (1959):
Chem. Abs., **53**, 9675 (25 May 1959)

A commercial pyrogene indigo $C_{36}H_{20}O_4N_4S_2{\cdot}2H_2O$ was dyed on cotton from a Na_2S bath at 80, 90, and 100°c.

The free energy of adsorption was calculated to be 3·71 cal. at 90°C. Heat of dyeing was 7·5 kcal. A dye molecule was accompanied by three Na cations on to the fibre. If a dyebath containing Rongalite and NaOH is used, then a dye molecule is accompanied by four Na cations with a free energy charge of 4·5 kcal. at 90°C. and a heat of dyeing of 9 kcal. C.O.C.

X-Ray Study of the Effect of Soaping on Cellulose dyed with some Anthraquinone Vat Dyes
J. O. Warwicker
J. Textile Inst., **50**, T 404–T 417 (June 1959)

X-Ray diffraction photographs of cuprammonium rayon heavily dyed with vat dyes show extra patterns due to the dye crystals. The diagrams are sharper, more intense and better oriented after the dyeings have been soaped, and therefore it is suggested that soaping promotes crystallisation of the dyes. The results are confirmed by comparing the above X-ray diffraction photographs with diffraction photographs of dope dyed cellulose acetate which almost certainly contains dye crystals. The implications of the results and the effect of the crystallisation of dye molecules within the fibre on its colour, are discussed. P.T.S.

Reactive Dyes
E. L. Caswell
Amer. Dyestuff Rep., **48**, 39–40 and 50 (18 May 1959)

The application of Cibacron reactive dyes to cellulose by the pad-steam, Thermofix, and package and beam dyeing methods, is discussed. The importance of removing unreacted dye is stressed. Cibacron dyes are applied to wool by starting dyeing in the presence of a non-ionic dispersing agent and acetic acid. After dyeing for 130 min. (75 min. at the boil), the pH of the dyebath is raised to 7 by the addition of hexamine and treatment is continued for 20 min. after which the material is rinsed. The material is finally treated for 10 min. at 70°F. with 2% formic acid.

Cibacrolan reactive dyes are applied to wool in the presence of a non-ionic agent and ammonium acetate. The material is aftertreated, in fresh baths, first with ammonia and then with formic acid. Blends of cotton and Dacron may be dyed continuously by padding the material through a liquor containing Cibacron dyes, selected disperse dyes, crystal gum and alkali, treating at 300°F. for 4–5 min., rinsing and finally soaping. W.P.M.

Package Dyeing of Blended Yarns of 80% Zefran plus 20% Cotton or Rayon
R. L. Burgess
Amer. Dyestuff Rep., **48**, 35–37 (18 May 1959)

Blended yarns of 80% Zefran acrylic fibre and cotton or rayon can be dyed with vat, azoic or sulphur dyes. The yarn is prepared for dyeing by scouring with an anionic detergent and tetrasodium pyrophosphate at 200°F. It may be bleached with sodium hypochlorite or acidified sodium chlorite. Selected vat dyes are applied by pigmentation followed by reduction using minimum alkali. For pale shades 2,2′-dihydroxy-5,5′-dichlorodiphenyl is used to retard the rate of dyeing of the Zefran. Higher temperatures (160–200°F.) are required to obtain heavy shades. The mode of application of azoic dyes is similar to the cotton method. Higher naphtholation temperatures cause partition in favour of Zefran while addition of salt has the opposite effect. Coupling times of the order of one hour are recommended. Sulphur dyes are dyed on to Zefran/cotton blends in the same manner as they are applied to cotton; but it should be noted that some sulphide complexes, recommended for dyeing sulphur dyes, discolour Zefran. Salt addition favours preferential build up on cotton while higher dyeing temperatures cause partition in favour of Zefran. W.P.M.

Methods of Assessing the Dyeing Properties of Wool Dyes
B. Kramrisch *J.S.D.C.*, **75**, 242–244 (May 1959)

Effect of a Surface Barrier on Uptake Rates of Dye into Wool Fibres
J. A. Medley and M. W. Andrews
Text. Research J., **29**, 398–403 (May 1959)

Any surface barrier to the entry of dye into a fibre exerts its greatest influence in the earliest stages of sorption, but becomes progressively less important as sorption proceeds. Samples of normal merino wool, abraded wool, and rough ground horn keratin membrane were used in experiments and, after allowing for variable diffusion characteristics of the fibres, a small effect was found, which could be due to the epicuticle. S.B.D.

Researches on Monolayers. VIII— Reactions of Metal-Unsulphonated Ligand (1 : 2) Dyes with Protein and Similar Monolayers and their relation to Dyeing Mechanisms
C. H. Giles and T. H. MacEwan
J.C.S., 1791–1798 (May 1959)

Dyes consisting of metal complexes with two unsulphonated azo cpd. as ligands are applied to monolayers of edestin and methoxymethylnylon, and their usual effects on the films are explained as due to absorption as aggregates. The complexes have a dichelate structure, and the aggregate, if ordered, must be a structure with "fins" of protruding aromatic nuclei, between which chain mol. in the film engage by non-polar, and in some cases polar, forces. A complex without pendant polar groups markedly increases film rigidity; proton-acceptor groups in a complex solubilise the films, and proton donor groups cross-link and stabilise them. The mechanism of dyeing of nylon and protein fibres is held to be adsorption (probably of micelles), (*a*) in water-accessible regions by ion-exchange, van der Waals forces, and, if suitable groups are available, hydrogen-bond donation from dye to backbone –CO·NH– groups; and (*b*) in water-inaccessible regions by van der Waals forces and by hydrogen bonding. Process (*b*) ("solid soln.") gives linear portions of adsorption isotherms. H.H.H.

Mechanism of Dyeing of Unlevel Dyeing Acid Dyes
M. Grindea, L. Coman, I. Bucurenciu, R. Zeidman, and T. Forst *Bul. inst. politechnic Iași*, **3**, 121–134 (1957): *Chem. Abs.*, **53**, 10769 (10 June 1959)

When applying acid dyes to wool the electrovalent bonds between dye and fibre are accompanied by van der Waals bonds in bad cases of unlevel dyeing. This is shown by formation of rings during neutral dyeing, this ring formation being independent of temperature. In acid baths these rings diffuse into the fibre probably as a result of labilisation of the van der Waals and H bonds between dye fibre. C.O.C.

Dyeing Wild Silk. XVII— Effect of Ash Content in Yamamai Silk on Dyeing
M. Mitsuishi and G. Aida
Shinsu-Daigaku Sen Igakubu Kenkyû Hôkoku, **7**, 161–165 (1957): *Chem. Abs.*, **53**, 7601 (25 April 1959)

Yamamai silk yields 1·24% ash. This was reduced to 0·16% and 0·07% by treating the silk by electrodialysis and with 0·0001 N-HCl respectively. On dyeing the silk with acid and basic dyes in a buffered bath more dye was absorbed by the silk of low ash content than by that of high ash content. Most of the inorganic compounds in yamamai silk are combined not with fibroin but with organic or inorganic in the silk. C.O.C.

Relationship to Dyeing Properties of the ζ Potential of Fibres treated with Some Metallic Ions
K. Kanamaru and S. Ota
Kôgyô Kagaku Zasshi, **60**, 611–615 (1957): *Chem. Abs.*, **53**, 7601 (25 April 1959)

Dynel, Acrilan, nylon, viscose rayon, and silk, swollen in 1% *p*-phenylphenol solution in NaOH at 100°C. for 15 min., were treated with 2–20% solutions of 1 : 2 : 2 mixtures of copper acetate (or chromium acetate or silver nitrate), KOH and dextrose at 100°C. for 1 hr. to add positivity to them. The ζ potential of treated fibres in water tends to be positive the stronger the solution. Variations in ζ potential with pH and pH with dyeing property were also studied. The results support the theory that the dyeing of these fibres with direct dyes is controlled mainly by electrostatic properties of the fibre surfaces, so that the dyeing characteristics of the fibres are improved by addition of positivity. C.O.C.

Causes of Uneven Dyeing of Sheep and Lamb Fur Skins
J. Jansó *Bőr és Cipőtech.*, **8**, 185–187 (1958): *Chem. Abs.*, **53**, 12720 (10 July 1959)

With formaldehyde-tanned skins the main causes of uneven dyeing are too high an ironing temperature and

uneven penetration of HCHO. They can be overcome by thermostatic control and use of wetting agents. In fat liquoring the main causes are uneven penetration (caused by drying too quickly or at too high a temperature) and use of unsuitable greases. Mineral and vegetable oils and nonionic emulsifiers did not cause uneven dyeing, but sulphonated oils did when dyeing browns and greys and soap when dyeing browns. Use of damp sawdust for tumbling is also detrimental. The fur must be sheared level before the formaldehyde treatment. To eliminate urine stains that develop in light and pastel dyeings on pannofix skins, the skins are given treatment with H_2O_2 and/or hydrosulphite before they enter the dyebath. C.O.C.

Level Dyeing of Furs
F. Brugger and L. Párizs
Bőr és Cipőtech., **8**, 188–190 (1958): *Chem. Abs.*, **53**, 12682 (10 July 1959)

Conventional dyeing usually results in the tips of the hair being lighter coloured than the rest. This is because the HCHO treatment imparts higher sp.gr. to the tips and so reduces their affinity for dyes, although the Cr uptake is uniform along the whole of the hair. Level dyeing with oxidation bases is best obtained if the H_2O_2 is added only after the CrO_3 has had time to react in the fibre with the oxidation base. The optimum time for adding the H_2O_2 is 60–90 min. before the end of dyeing. If this is done level blacks can be produced and 20–30% saving in oxidation base is effected. C.O.C.

Dyeing Synthetic Fibre. II— Dyeing Nylon with Suspensions of Acid and Mordant Dyes
V. F. Androsov and A. A. Kharkharov
Izvest. Vysshikh Ucheb. Zavedenii, Tekhnol. Tekstil. Prom., (6), 62–65 (1958): *Chem. Abs.*, **53**, 12682 (10 July 1959)

The water-insoluble Ca, Mg or Ba salts of acid and mordant dyes when used as disperse dyes yield level dyeings on nylon which are equally as fast as those dyed by conventional acid and mordant dye methods. The time required for dyeing is less. C.O.C.

Diazo Reaction in the Coloration of Hydrophobic Man-made Fibres
H. R. Hadfield and W. F. Liquorice
J.S.D.C., **75**, 299–307 (June 1959)

Dyeing and Finishing Blends of Dacron Polyester Fibres and Cellulose Fibres
J. J. Iannarone, R. J. Thomas, and C. W. Holweger
Amer. Dyestuff Rep., **48**, 41–45 (4 May 1959)

The preparation of blends of "Dacron" type 54 and cotton for dyeing is similar to that for cotton fabrics except that the grey singe and kier boil are omitted and the fabric is set or crabbed in open width. Disperse dyes may be applied at the boil with carrier or at high temperature. Carrier methods are generally used for "Dacron" fabrics but increased yardages may lead to the greater use of the "Thermosol" process. Selection of disperse dyes, which is dependent upon many factors, is discussed. Vat dyes are recommended for production of optimum fastness on the cellulose fibre and may be applied by either pad–steam or pad–jig methods. "Dacron" type 64 is susceptible to hydrolytic degradation in hot liquors and sodium sulphate is added to all hot aqueous baths to prevent it. Cationic dyes are applicable to "Dacron" type 64 to produce bright shades for ladies' wear as well as economical black and navy shades. The influence of finishing techniques such as setting, singeing, calendering, mechanical shrinking, and resin and waterproof finishes, on the appearance and wash and wear properties of the fabrics, is discussed. W.P.M.

Dyeing of Polyester Fibres with Disperse Dyes. Mechanism and Kinetics of the Process for Purified Dyes
D. Patterson and R. P. Sheldon
Trans. Faraday Soc., **55**, 1254–1264 (July 1959)

The system polyethylene terephthalate fibres + water + disperse dye is used to investigate the kinetics of the dyeing process. Solubilities of dyes in water and in the fibre and desorption rates from dyed fibres are determined. Heats of dyeing, diffusion coefficients, and activation energies are derived. The mechanism of dyeing is postulated as dissolution of dye molecules in water followed by diffusion of single dye molecules inside the fibre structure. A rate equation for dyeing is proposed in terms of diffusion coefficients and an attempt is made to relate the latter to structural differences between fibres. W.R.M.

Hydroxylamine in the High-temperature Dyeing of Acrylic Fibres
F. Schouteden J.S.D.C., **75**, 309–310 (June 1959)

Recent Advances in the Dyeing of Acrilan Acrylic Fibre
E. V. Burnthall
Amer. Dyestuff Rep., **48**, 41–45 (18 May 1959)

The absorption of dyes by acrylic fibres is discussed. The absorption of basic dyes by Acrilan is largely pH dependent and increases with rise in pH to a maximum at approximately pH 8. Ammonium acetate, recommended as dyeing assistant for dyeing basic dyes on Acrilan, enables only medium-heavy depths to be obtained because the rise in pH of the dyebath during dyeing is insufficient for maximum colour yield. The use of methyl alcohol instead of acetic acid in dissolving the dyes gives better results; but urea is much more effective. Using urea the pH of the dyebath rises steadily from pH 5·5 to 7 during the dyeing period and tinctorial yield is 30–40% greater. Increase of urea concentration increases the rate of dyeing. Some non-ionic surface-active agents contribute to build up and have no effect on fastness. The use of urea when dyeing Acrilan/rayon blends causes reduced staining of the rayon and enables the dyeing time to be reduced. The addition of urea to print pastes enables prints of very high colour value to be obtained with basic dyes. W.P.M.

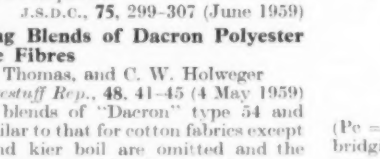
Fluorescent Brightening
W. Wincor *Textil-Rund.*, **14**, 316–328 (June 1959)

A survey of the development, properties, assessment, and fields of application of the compounds used. S.R.C.

PATENTS

Colouring Cellulosic Textiles with Solubilised Phthalocyanine Dyes
ICI *BP* 816,925

Dyes of formula—



(Pc = phthalocyanine, Y = direct bond or a divalent bridging radical and the system (Pc)–Y– contains at least one solubilising group; R = H, Alk, aralkyl or cycloalkyl; A = Cl or O–B (B = subst. or unsubst. hydrocarbon); n = a whole number), prepared e.g. according to *BP* 805,562 (J.S.D.C., **75**, 168 (March 1959)) are applied to cellulosic textiles in an aqueous solution containing > 3% of an acid binding agent. Thus cotton cloth is padded with an aqueous solution containing a turquoise blue phthalocyanine dye, together with soda, Glaubers salt, and a highly sulphonated oil. It is then heated for 5 min. at 110°c., rinsed, soaped at the boil, rinsed and dried to yield a bright turquoise blue fast to washing and light. The dye used was obtained by treating 1 mol. of copper phthalocyanine tetra-3-sulphonchloride with an aqueous soln. of 2 mol. Na-2,5-diaminobenzene sulphonate and treating the resulting product in aqueous solution at < 5°c. with one-third of its weight of cyanuric chloride. C.O.C.

Dyeing Cellulosic Textiles with Reactive Dyes
ICI *BP* 819,585

Modification of *BP* 797,946 (J.S.D.C., **74**, 714 (1958)). Applying the acid-binding agent before the dye yields colorations of high fastness to wet processing and avoids the need to manufacture alkali cellulose. Thus cotton is padded to pick up twice its weight of 1% aq. NaOH and dried at 100°c. It is then similarly padded with an aqueous solution containing 2% of the red dye described in Example 5 of *BP* 779,946, dried and steamed for 1 min. at 100°c. Finally it is rinsed, residual alkali partly removed in aqueous sodium carbonate, soaped, boiled, rinsed, and dried. This yields a bright red fast to washing and light. C.O.C.

Aromatic Monocarboxylic Esters as Assistants in Dyeing Hydrophobic Fibres
American Cyanamid Co. *USP* 2,881,045

Emulsions of aromatic carboxylic esters of any type, e.g. methyl salicylate and methyl ester of 3-hydroxy-2-naphthoic acid, added to any type of dyebath, e.g. disperse, acid, sulphur or vat dyebath, result in much heavier dyeings being obtained. The same effect is obtained in azoic dyeing, application of pigments or fluorescent brightening agents. Thus addition of an aqueous emulsion of methyl salicylate to a dyebath containing C.I. Disperse Red 1 resulted in a deep scarlet dyeing whereas in absence of the methyl salicylate only a commercially unsatisfactory, dull and spotty dyeing was obtained. C.O.C.

Butyl Benzoate as Assistant in Disperse Dyebaths
Celanese Corpn. of America *USP* 2,880,050

Presence of 10% (on the weight of the fibres) of butyl benzoate in disperse dyebaths greatly increases the rate of dyeing, the degree of exhaustion and the fastness to rubbing of the resultant dyeings. C.O.C.

Dyeing Polyesters in High Temperature, Non-aqueous Disperse Dyebaths
DuP *USP* 2,882,119

Decomposition of disperse dyes in non-aqueous liquids at > 130°C. is prevented if the bath contains 0·1–0·5% of H_2SO_4, H_3PO_4, monochloroacetic, tartaric or oxalic acid and ethylene glycol, di- or triethylene glycol, propylene or dipropylene glycol. Thus in the continuous dyeing of Terylene fabric with the blue dye 1,8-dihydroxy-4-anilino-5-nitroanthraquinone from a diethylene glycol bath at 180°C. good level dyeings are obtained at the start but after 1 hr. the dyeings obtained become steadily duller, greener, and weaker. If phosphoric acid 85% (0·4% on the bath) is added to a fresh bath then this deterioration in the dyeings obtained from it does not occur. C.O.C.

Increasing the Wash Fastness of Disperse Dyes on Cellulose Acetate
Celanese Corpn. of America *USP* 2,880,052

Cellulose acetate or other organic derivative of cellulose is dyed with a disperse dye containing a reactive group, e.g. the blue 1,4-di(hydroxyethylamino)anthraquinone and the red 4-nitrophenyl-azo-4′-bis(β-hydroxy ethyl)-aminobenzene, treated with a soluble amidogen-form-aldehyde precondensate, e.g. *NN′*-dimethoxymethyl ethylene urea, and finally baked. The resultant dyeing is fast to washing at 120–130°F. C.O.C.

Synthetic Resin Binder Compositions for Pigments (III p. 549)
Pigment-binding Composition (III p. 549)
Affinities, Heats of Dyeing, and Entropy Changes of Some Blue Direct Dyes (IV p. 522)
Disperse Dyes. XVI — Dyeability and Fastness of Aminoanthraquinones (IV p. 555)
Natural Dyes and their Use in Dyeing Foods (IV p. 558)
Enhancing the Affinity of Vinylidene Cyanide Copolymer Fibres for Acid Dyes (VI p. 567)
Pigmented Prints, Impregnations and Coatings on Fibrous Materials (IX p. 572)
Amidoximation of Partly Cyanoethylated Cotton Cloth. Its Effect on Affinity for Acid and Basic Dyes (X p. 573)
Colouring Cheeses with Annatto (C.I. Natural Orange 4) and Carotene Dyes (C.I. Natural Yellow 26) and the Detection of Such Dyes (XIV p. 580)

IX— PRINTING

Application of Remazol (FH) Dyes in Printing
H. Zimmermann
Melliand Textilber., **40**, 539–544 (May 1959)

A detailed discussion, with numerous recipes, of direct and discharge printing with Remazols. Stability of printing paste varies from 1–2 days for Brilliant Orange RR and Red B to several weeks. Aftertreatment with chelating agents causes shade changes and lowering of light fastness with Yellow RT, Red 3B, and Red-violet R. This is remedied by aftercoppering. Fastness properties are tabulated, as is also the effect of various resin treatments on light fastness. Melamine-formaldehyde resins cause some and reactant-resins appreciable reduction. The light fastness is improved somewhat on washing. Some trials with zinc catalysts behaved anomalously, giving good results on Fade-Ometer, but poor ones on daylight exposure. S.M.J.

Practical Direct Printing with Pigments
H.-W. Jung *Melliand Textilber.*, **40**, 536–539 (May 1959)

An informative and concise review of practical experience concerning pigment printing, useful in the printworks. Resin binders, emulsions, thickenings, storage, stability, and fixation are discussed. S.M.J.

Elucidation of the Mechanism of Colour Development by Investigation of the Kinetics of the Reaction
J. Eggers
Wiss. Phot., Intern. Konf. Köln., 471–476 (1956): *Chem. Abs.*, **53**, 6847 (25 April 1959)

Oxidation products of *p*-aminodialkylanilines were added in solution to *p*-aminodialkylanilines and the kinetics of the reaction of this solution with couplers in absence of oxidants were followed. The reaction kinetics of oxidation of *p*-aminodialkylanilines at low alkalinity were determined and colour formation initiated by adding couplers after definite oxidation times was followed. Results indicate that the fully oxidised form of *p*-aminodialkylanilines is the reactant in dye formation. C.O.C.

Relationship between the Optical Density of Silver and Dye in Colour Development
Wiss. Phot., Intern. Konf. Köln., 485–493 (1956): *Chem. Abs.*, **53**, 6847 (25 April 1959)

The relationship between the optical densities of dye and Ag in colour development with diffusing couplers was determined for several couplers with diethyl-*p*-phenylenediamine as developer. The optical density of the dye was measured in the maximum of the spectral absorption, that of the Ag was determined after removing the dye from the photographic image. Dye density was proportional to Ag density, the relationship being independent of the duration of development. C.O.C.

Colour Development of Photographic Materials for X- and γ-Rays
W. Eichler
Wiss. Phot., Intern. Konf. Köln., 596–598 (1956): *Chem. Abs.*, **53**, 6848 (25 April 1959)

Colour development of X-ray films coated on both sides of the support is used to increase the scale of ds. which can be resolved by the eye. C.O.C.

Kinetics of Colour Development of Multiple Layer Fibres as a Function of Temperature
Y. N. Gorokhovskiĭ
Wiss. Phot., Intern. Konf., Köln., 494–497 (1956): *Chem. Abs.*, **53**, 6847 (25 April 1959)

The mutual influences of the layers upon the kinetics of colour development of multilayer film depends very much on the temperature. Transfer of developer components by diffusion from one layer to another increases with increasing temperature and development time, and with decreasing thickness of the individual film layer. C.O.C.

Some Problems of Sensitometry of Multilayer Colour Materials
Y. N. Gorokhovskiĭ
Wiss. Phot., Intern. Konf. Köln., 526–529 (1956)

Chemistry of Colour Developers: Effect of Ethylenediaminetetraacetic Acid and Iron on Loss of Developing Agent and Sulphite from Aerated Solutions
P. M. Mader *Phot. Sci. & Eng.*, **3**, 49–56 (1959)

PATENTS

Illuminated Discharge Printing on Synthetic Polymer Fibres
Hardman & Holden *BP* 816,930

Illuminated discharges are obtained on dischargeable dyes on polyester, polyamide, and acrylic fibres by use of a discharge paste containing 2–4% of thiourea dioxide (C.I. Reducing Agent 11) and an anthraquinone disperse dye containing no azo groups. Addition of 0·5–1·0% of 1 : 1 aq. ammonium nitrate to the paste accelerates reoxidation of the reduced dye without impairing the reducing properties of the thiourea dioxide. Alternatively the reducing paste can be printed on first followed by application of a dischargeable dye as the ground colour. C.O.C.

Pigmented Prints, Impregnations and Coatings on Fibrous Materials

ICI *BP* 816,985

Use of a thermosetting resinous precondensate and a polymer derived from butadiene, an acrylic or methacrylic amide and an acrylic or methacrylic ester or nitrile, as the binder yields prints, etc., which have excellent fastness to severe washing and rubbing and also an exceptionally soft handle. Thus an aqueous dispersion containing a butylmethacrylate–acrylamide copolymer and a butadiene–acrylonitrile polymer is thickened with white spirit and mixed with an aqueous paste of dimethylolurea and copper phthalocyanine (C.I. Pigment Blue 15) with NH_4 thiocyanate as catalyst. When the mixture is printed on to viscose staple fibre, dried and baked for 4 min. at 130°c. a supple print having good fastness to washing and rubbing is produced. C.O.C.

Flock Patterns on Textiles

Heberlein & Co. *BP* 816,354

Cloth which is at least partly covered with a layer of short textile fibres or a fine powder secured to it with an adhesive has applied to it a composition which coalesces or fuses the layer where it is applied. This results in the areas to which the composition has been applied becoming smooth. The composition must not affect the adhesive. Thus cotton cloth coated with viscose rayon flock using natural rubber as adhesive is printed on with a paste of $ZnCl_2$ (69 g.), water (31), and viscose rayon waste (2). It is then dried at 90°c., washed with cold water and dried under tension. C.O.C.

Transfer Papers

Hoffmann & Engelmann *BP* 819,695

The base paper is first coated with starch and then with polyethylene oxide waxes. It has the advantage of permitting transfer either by moisture or heat of the design printed on it. C.O.C.

Transfers

S. W. Cox *BP* 819,573

A flexible base is coated with a powder or granular substance to be transferred using as binder a synthetic thermosetting resin. C.O.C.

Diazotype Material

General Aniline *BP* 815,956

Clean, bright prints fast to washing and rubbing are obtained from diazotype material comprising a base coated with finely-divided non-colloidal silica dispersed in an alkali-soluble, water- and acid-insoluble vinyl compound—ethylene-$\alpha\beta$-dicarboxylic acid or anhydride or ethylene monocarboxylic acid, this coating being subsequently sensitised with a diazonium compound and a coupling component. C.O.C.

Photographic Iron–Silver Colour Process

Panacolor *USP* 2,886,435

Film is sensitised with aqueous ferric ammonium oxalate exposed through one set of colour separation negatives, and treated with aq. $AgNO_3$ to produce a silver image. The silver image is then bleached into a silver salt image and treated with a colour developer in presence of a coupler to produce a dye image and finally the silver is removed. This sequence of steps can be repeated several times in order to obtain multicoloured images. C.O.C.

Synthetic Resin Binder Composition for Pigments (III p. 549)
Pigment-binding Composition (III p. 549)
Mordants for Acid Dyes used in Photography (III p. 550)
Solvents for Colour Couplers (III p. 550)
Interdependence of Colour and Structure of Some Dyes formed in Colour Development (IV p. 554)
Colouring Cellulosic Textiles with Solubilised Phthalocyanine Dyes (VIII p. 570)
Rendering Polyethylene Surfaces Adherent to Hydrolysed Vinyl Chloride–Vinyl Acetate Copolymers, Polyvinylalkyl Ethers, and Acrylic or Methacrylic Polyesters (XIII p. 577)
Spectrophotometric Determination of Dyes in Hydrophobic Colour Images (XIV p. 579)

X—SIZING AND FINISHING

Textile Finishing in the Early 1900s

H. à Brassard J.S.D.C., **75**, 310 (June 1959)

Physicochemical Characteristics of the Wetting of Textiles by Solutions of High-molecular Compounds

V. E. Gul, R. Y. Fainberg, M. G. Maizel's, V. G. Raevskiĭ, and M. I. Sin'kova

Izvest. Vysshykh Ucheb. Zavedenii, Khim. i Khim. Tekhnol., (5), 114–119 (1958): *Chem. Abs.*, **53**, 9681 (25 May 1959)

The effect of viscosity (η) on the kinetics of the change of the boundary wetting angle (θ) is characterised by τ_{max}, i.e. time necessary for a drop to become stationary. The value of τ_{max} expresses the combination of all processes occurring between cloth and glue and serves as a criterion in the evaluation of the degree of receptivity of any textile material for any glue. From the curves θ–τ for glues of different η it is seen that τ_{max} is the smaller the less the viscosity. These curves show that in spite of a decrease in viscosity and surface tension, introduction of polar compounds retards the variation of θ with time and correspondingly the value of τ_{max} in that case sharply increases. Photographs show that in placing a drop of glue with added alcohol on Perka B (a cotton cloth), the diameter of the drop remains constant; this indicates sharp slowing down of the flowing process. The greater the rate of variation of θ and the less τ_{max} the greater the stability of the bond between the glue and the fibres. All cotton cloths have greater adhesive power for the various rubber coatings and so they can be rubberised much more quickly than can cloths made from synthetic fibres. C.O.C.

Cellulose and Formaldehyde

J. T. Marsh J.S.D.C., **75**, 244–252 (May 1959)

Sorption of Formaldehyde by Cellulose from Aqueous Solution

W. J. Roff *J. Textile Inst.*, **50**, T 353–T 372 (May 1959)

The sorption of formaldehyde by cellulose has been followed by measuring the fall in concentration of an aqueous formaldehyde solution after cellulose has been introduced. The reaction is reversible and the formaldehyde can be completely removed by rinsing in water for a sufficiently long time. The changes in physical properties of regenerated cellulose (wet strength, birefringence) which accompany formaldehyde sorption are also fully reversible by rinsing. The activation energy of the forward reaction is 13 kcal. per mole. The equilibrium formaldehyde uptake of different cellulose samples is related to their moisture regain at 65% R.H. The equilibrium uptake of formaldehyde is approximately proportional to the square root of its concentration in the solution. Methylolcelluloses are probably formed. P.T.S.

Chemically Modified Cotton

K. V. Usmanov, V. I. Sadovnikova, and G. M. Kozin

Uzbek. Khim. Zhur., Akad. Nauk Uzbek. S.S.R., (2), 21–28 (1958): *Chem. Abs.*, **53**, 7585 (25 April 1959)

An account of the treatment of cotton with such monomers as acrylonitrile, styrene, isobutylene, etc. Study has been made of the best reaction conditions for the cyanoethylation of cotton. C.O.C.

Use of Polyethylene Emulsions in Textile Applications

R. Rosenbaum

Amer. Dyestuff Rep., **48**, 46–49 (18 May 1959)

Polyethylene can be emulsified in non-ionic, cationic, or anionic systems. The non-ionic emulsions are most widely used because they are compatible with most of the common textile finishing agents and do not yellow, change the shade of dyestuffs or retain chlorine. Small amounts of deposited polyethylene cause a high increase in tear strength of fabrics made from natural and synthetic fibres and in blended fabrics the flex abrasion and tear strength are improved. In conjunction with thermosetting resins the polyethylene gives higher crease-resistance improvement over that imparted by the resin alone and minimised reduction in tear strength and flex abrasion. A properly formulated emulsion is non-yellowing, scorch

resistant and will impart improved handle. The polyethylene remains in the fabric even after twenty launderings. W.P.M.

Coating Fabrics, especially with Chlorosulphonated Polyethylene

D. Eisele *Melliand Textilber.*, **40**, 84–86 (Jan. 1959)

Qualitative tabulation of resistance to weathering, cold, heat, fire, oil, and abrasion purports to show the superiority in general of chlorosulphonated polyethylene in fabric coating to elastomers based on 2-chlorobutadiene, butadiene-acrylonitrile, isobutene-isoprene, and polyurethane. Hypalon (DuP) is made by reacting a soln. of high pressure polyethylene (mol.wt. ca. 20,000) with Cl_2 and SO_2 to give $[-(-CH_2{\cdot}CH_2{\cdot}CH_2{\cdot}CHCl{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2-)_{18}-CHSO_2Cl]_{17}$. It can be vulcanised with accelerator and (1) metal oxide and organic acid, (2) metal oxide only, (3) epoxy resin. (1) Probably reacts according to—

$$2RCOOH + PbO \rightarrow (RCOO)_2Pb + H_2O$$
$$2RSO_2Cl + 2H_2O \rightarrow 2RSO_2OH + 2HCl$$
$$2HCl + PbO \rightarrow PbCl_2 + H_2O$$
$$2RSO_2OH + PbO \rightarrow (RSO_2O)_2Pb + H_2O$$

$$2RCOOH + 2RSO_2Cl + 3PbO \rightarrow (RSO_2O)_2Pb + (RCOO)_2Pb + PbCl_2 + H_2O$$

(2) relies on atmospheric moisture, requires less metal oxide, but more accelerator, and is more controllable. In (3) the resin accepts HCl and more accelerator is used. This vulcanisate has low specific weight, and is highly stable, except towards direct sunlight unless pigmented by the ultraviolet-absorbing carbon blacks. Coatings are applied by calendering or as organosols. Calendering requires 3 layers, the first in particular containing adhesives and softeners and the major second one low mol.wt. polyethylene. Adhesion to synthetic fabrics is provided by pretreatment with isocyanates, either in soln., alone or with chlorosulphonated polyethylene in the absence of all moisture, or blocked, below 150°C. by phenol in a 2-chlorobutadiene latex. Organosols of 40–60% dry weight are prepared by adding other ingredients, dispersed by 2 days' ball milling, to the Hypalon soln. Vulcanisation can be initiated with infrared heaters at 180°C. Hypalon 30 contains more Cl and resists oil better. S.M.J.

Organosilicon Compounds in Cotton Finishing

P. A. Simigin

Khim. i Prakt. Primenenie Kremnemg. Soedinenii, Trudy Konf., Leningrad, (4), 49–54 (1958): *Chem. Abs.*, **53**, 11847 (25 June 1959)

Equimolecular mixtures of $CH_3Si(OCOCH_3)_3$ and $(CH_3)_2Si(OCOCH_3)_2$ (I) and the following: $CH_3Si(OCOCH_3)_2OCOC_{17}H_{35}$ (II), $[(CH_3)_3SiO\cdot Si(OCOCH_3)O]_n$ (III), $(C_2H_5Si(H)OSi(CH_3)_2O)_n$ (IV), $(C_2H_5SiHO)_n$ (V) and $(C_2H_5SiHNH)_n$ (VI) were used on cotton apparel and tent cloths. With I, II, III, and IV the solvent was CCl_4 and the catalyst $Ti(OC_2H_5)_4$. V was used in aqueous emulsion and VI dissolved in CH_2CHCl_2. Before the silicone treatment the cloths were boiled or dyed. In each case the samples were given two immersions in a 5% solution of the silicone and were air dried at < 80°C. and then baked at 160°C. for 8 min. All the treatments resulted in better washability and quicker drying than did an aluminium–paraffin wax–stearic acid finish but the latter imparted better hydrophobic properties. II gave greatest improvement in washability. VI, though causing more damage to the cloth than I, II, III, IV, and V, gave better weatherproofing. In a 5-month exposure to light and weather all the silicone-treated samples suffered as much damage as an untreated sample. The silicone treatments resulted in better crease shedding and dimensional stability and only slightly lowered the air porosity. It is concluded that the silicones, particularly II, are suitable for water-repellent finishing of cloths not subject to continuous exposure to light and weather but that use of organic solvents presents many difficulties in works practice. C.O.C.

Antimonites in Flame-resistant Finishes

M. P. Khera, R. M. Desai, and M. G. Bhargava

J. Sci. Ind. Research (India), **17A**, 452–454 (1958): *Chem. Abs.*, **53**, 8636 (10 May 1959)

Successive impregnations are given with (*a*) alkaline solution of Na or K antimonite, (*b*) aq. NH_4 or Na acid phosphate and (*c*) chlorinated rubber solution or dispersion. (*a*) Imparts flame resistance, (*b*) glow resistance and (*c*) renders the finish fast to washing. Strength, air, and water permeability remain unaffected but the fibres become stiffer. A certain degree of resistance to attack by microorganisms is also given. C.O.C.

Amidoximation of Partly Cyanoethylated Cotton Cloth. Its Effect on Affinity for Acid and Basic Dyes

M. Negishi and H. Ito

J. Soc. Textile Cellulose Ind., Japan, **15**, 116–123 (Feb. 1959)

Study of the amidoximation of cyanoethylated (3–5% N) cotton cloth with aq. $NH_2OH{\cdot}HCl$. A neutral or slightly alkaline solution of NH_2OH at 80°C. is needed. Addition of non-ionic surfactants slightly slows down amidoximation. Amidoximation, especially low amidoximation of lower cyanoethylated cotton and the slow raising of the reaction temperature impart considerable increase in crease and flexabrasion resistance and regain while little affecting tensile strength, elongation, tearing strength, and softness of handle. Even a lower degree of amidoximation gives striking increase in affinity for acid dyes and great decrease in affinity for basic dyes. C.O.C.

Formation of Vinyl Polymers on Wool

G. Prati *Ann. chim.* (Rome), **48**, 1057–1069 (1958): *Chem. Abs.*, **53**, 11846 (25 June 1959)

Wool is steeped in 0·1% aq. $FeSO_4$ for 4 hr. at room temp. and then treated in an aqueous emulsion of $CH_2{:}C(CH_3)COOCH_3$ (0·19 M.), H_2SO_4 (0·02 M.) and a polyethylene oxide–octyl phenol condensate at 50°C. Samples were first treated with acetic anhydride at 100°C., methanol containing 5% N-HCl, 2·5% K acid tartrate + 3% $K_2Cr_2O_7$, 3 g./l. HCHO or 0·1 N-$HSCH_2COOH$. Except in the last case, the polymer-coated fibre has good physical properties. The neutral wetting agent penetrates the positively changed wool (in acid solution) better than a cationic wetting agent. Wool reduced by $HSCH_2COOH$ is weakened by the polymerisation, a low degree of polymerisation also weakens the fibre. C.O.C.

Setting Reduced Keratin Fibres with 1-Fluoro-2,4-dinitrobenzene

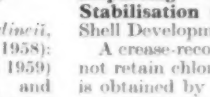
P. T. Speakman J.S.D.C., **75**, 252–254 (May 1959)

PATENTS

Imparting Crease-recovery and Dimensional Stabilisation to Textiles with Polyepoxides

Shell Development Co. *USP* 2,886,472

A crease-recovery and shrink-resistant finish which does not retain chlorine and has excellent resistance to washing is obtained by use of aqueous solutions or dispersions of polyepoxides containing at least one—



group, e.g. vinyl cyclohexane dioxide, bis(epoxycyclohexyl)propane and epoxydised tetrahydrobenzyl tetrahydrobenzoate.

USP 2,886,473

Use for the above purpose of a mixture of a polyether polyepoxide, rubber and an epoxy curing agent results in the finished fabric having good tensile and tear strength. C.O.C.

Treating Light, Loosely-woven Cotton or Spun Rayon Fabrics with Amino-formaldehyde Resins and Rubber

E. Elod *BP* 816,330

Low grade, light, loosely woven cotton or spun rayon fabrics are impregnated with aqueous dispersions containing rubber and aminoformaldehyde precondensates containing methylol groups which can react with the rubber and then baked. The weight of precondensate present must be more than that of the rubber. The treated fabric is non-adhesive and has much improved handle, elasticity, and resistance to creasing. It is also resistant to ageing and dry cleaning.

BP 816,506

Treatment with resins alone is claimed. C.O.C.

Coating Shaped Polymers with a Non-polymerisable Compound

DuP *BP* 816,024

The shaped polymer is exposed to ≤ 1 watt-second per sq.cm. of surface of ionising radiation with an energy of

< 0·1 million ev. and then, in absence of radiation and while the polymer's surface is still activated, treated with a fluid form of a compound which is chemically distinct from the polymer, non-polymerisable and fluid at a temperature below the softening point of the polymer. This forms a coating which remains united with the polymer even when treated with solvents which normally dissolve the compound used for the coating. Thus polyester film is irradiated and then treated with molten polyethylene oxide of mol.wt. 20,000. After being treated for 20 hr. with ethanol in a Soxhlet extractor the film has better affinity than an untreated control for disperse and vat dyes. Nylon and cotton cloths when irradiated and then treated with $CH_3OCH_2CH_2OH$ at − 78°c., placed in an evacuated sealed tube for 16 hr. at room temperature, extracted for 5 hr. in a Soxhlet with acetone and then direct under vacuum for 15 hr. at 100°c. still contained 0·02% of the β-methoxymethanol. C.O.C.

Reducing the Tendency of Wool to Felt
Boehme Fettchemie *BP* 818,162

Treatment with compounds, preferably fatty or resinous, containing epoxide groups as well as with oxidising agents is a quick method of obtaining an improved shrink-resisting finish. Thus 1 mol. disodium phthalate and 2 mol. epichlorohydrin are reacted to give a resin. This resin (50 g.) is dissolved in tetrahydrofurfural alcohol (200 c.c.) and the addition product (25 g.) of 5 mol. ethylene oxide and a C_{12}–C_{14} mixture of aliphatic alcohols is added. The solution (5 c.c.) and glacial acetic acid (12 c.c.) are strained into cold water (1000 c.c.). To the resulting emulsion there is added Na hypochlorite (to yield 0·3 g./l. of active chrome). Knitted woollen fabric is treated in this liquor for 5 min. and a liquor ratio of 1 : 30. It is then dechlorinated with $NaHSO_3$, rinsed and dried at 70°c. The treated fabric shows no reduction in whiteness and after repeated washing shows no shrinkage and has a full, soft handle. C.O.C.

Reducing the Alkali Solubility of Wool by Treatment with Acrolein
Fabric Research Laboratories *USP* 2,880,055

Wool treated with an 0·3–6·0% aqueous solution of acrolein at pH 2–7 and, preferably at > 65°c. has its resistance to damage by treatment under acidic, oxidising or reducing conditions much increased. The process also reconditions wool that has already been damaged by such treatment. C.O.C.

Treating Wool with Organic Diisocyanates in Presence of a Tertiary Amine
U.S. Secretary of Agriculture *USP* 2,880,054

Wool is cross-linked by treating it under anhydrous conditions at 20–120°c. with an organic diisocyanate in presence of a tertiary amine, preferably pyridine. The amine may be used in excess so as to act both as catalyst and solvent for the diisocyanate. The treated wool displays decreased supercontraction compared with untreated wool and has also a greatly reduced tendency to felt and greater resistance to alkali and acid. The tensile strength, handle, and colour are not affected. C.O.C.

Treating Protein Fibres with Dicarboxylic Acid Diazides
U.S. Secretary of Agriculture *USP* 2,881,046

Wool treated with a dicarboxylic acid diazide contracts much less than untreated wool when treated with a reducing agent capable of splitting disulphide bonds. In some cases, particularly with succinazide, the treated wool has reduced felting properties. The treatment also increases resistance to acids and alkalis but does not adversely affect tensile strength, handle, or colour. C.O.C.

Surface Saponification of Cellulose Ester Fabrics
Celanese Corpn. of America *BP* 815,794

Fabric formed from cellulose ester fibres containing > 0·25 alcoholic hydroxyl group per anhydro glucose unit is treated with strong aqueous alkali so as to saponify only the surfaces of the fibres. Thus fabric of cellulose acetate of acetyl value > 61% is treated with aq. 20% NaOH at 20–40°c. and is then well rinsed. This completely saponifies the surface of the fibres while the remainder remains intact. C.O.C.

Stiffened Cellulose Triacetate Fabrics
BrC *BP* 816,238

Cellulose triacetate fabrics when impregnated with a heat-hardenable resinous precondensate and then baked are very useful as interlinings for the collars and cuffs of shirts. They can be set to form by heat and retain this form on being washed. C.O.C.

Treating Polyester Fibre Fabrics with Alkali
Heberlein & Co. *BP* 818,121

When treating polyester fibre fabrics with aqueous alkali metal hydroxide addition of 10–30% by weight of the bath of an organic swelling agent and/or solvent (which is at least partly soluble in water) enables the time of treatment to be so shortened that it can be made into a continuous process. Thus Terylene fabric is passed continuously in open width through a solution made up of NaOH 38° Bé (15 parts by volume), diethylene glycol 21·2% by weight (5) and *n*-butanol 3·1% by weight (1) at 100°c. The fabric is in the liquor for 45 sec. and after being squeezed is rinsed with water, neutralised with acetic acid, rinsed and dried under tension. It loses 15% in weight and acquires a pleasant, soft handle. C.O.C.

Treating Tubular Knit Fabric with Resins
Samcoe Holding Corpn. *USP* 2,880,114

The fabric is spread out flat and steamed while maintained at the finished length and width. It is then fed, still spread out, to a pair of squeeze rollers, then into a bath of resin precondensate, through a second pair of squeeze rollers and then dried and baked. Suitable machinery is described. C.O.C.

Imparting a Springy Handle to Nylon, Acrylic and other Nitrogenous Hydrophobic Fibres
American Cyanamid Co. *USP* 2,887,408

Treatment with an aqueous solution or suspension of a precondensate of HCHO with a compound of formula $S((CH_2)_nCONH_2)_2$ (n = 0–4) and an acid catalyst followed by drying and baking is used to produce a springy handle without increase in flammability. Thus a 1 oz. nylon twill was slop padded with a solution of water (65·45), NH_4Cl (1·25), and a 75% solids commercial flame retardant finish for nylon (33·3). The flame resistant finish contained partly methylated thiourea–formaldehyde and partly polymerised urea–formaldehyde resin. The treated fabric was then run through a microset pneumatic padder so that it had a resin solids content of 20% of its dry weight. Finally it was dried and cured in one operation for 6 min. at 290°F. It had a stiff springy handle and its flammability was identical with that of the untreated fabric. C.O.C.

Antistatic Treatment of Nylon or Polyethylene Terephthalate Fibres
ICI *BP* 817,355

The fibres are treated with a condensate which is liquid below 30°c. of 1–3 mol. fatty acid and 1 mol. of glycerine previously condensed with 3–40 mol. of ethylene oxide. Thus a Terylene staple fibre fabric was padded with an aqueous solution of the condensate of 1 mol. lauric acid and 1 mol. glycerine previously condensed with 6 mol. ethylene oxide. The take-up of the condensate was 0·2% of the weight of the fibre. After drying the fabric had excellent antistatic properties. C.O.C.

Bonding Polyethylene Terephthalate Fibres to Rubber
ICI *BP* 816,640

Polyethylene terephthalate fibres after being treated with 4,4′-diisocyanato diphenyl methane or 4,4′,4″-triisocyanato diphenyl methane are readily bonded to natural or synthetic rubber by an adhesive based upon a butadiene–vinyl pyridine latex and a resorcinol–formaldehyde solution. C.O.C.

Reducing Pilling
Wool Industries Research Assocn. *BP* 819,747

One side only of the fabric is given a shallow impregnation of butadiene or butadiene–acrylonitrile latex so that the polymer adheres to those fibres at or near the surface and prevents them migrating. C.O.C.

Down-proof Finish
BrC *BP* 816,511

Fabric whose weave is not close enough for it to be down-proof is rendered down-proof by spraying it with poly(vinyl acetate) or other thermoplastic polymers of

mono-olefinically unsaturated compounds so that the coating is not a film but is made up of fine particles of the polymer. C.O.C.

Stretchable Hose
Patentex *BP* 813,742

Ladies' stockings of improved elasticity are produced by simultaneous knitting of a twisted lively yarn and a non-lively untwisted yarn. The lively yarn is produced by twisting, heat-setting, and back-twisting. The hose are finished by steaming at 210°F., drying, dyeing, and post-boarding in steam at 250–255°F. G.E.K.

Impregnated Cellulosic Materials for Preserving Metals Susceptible to Atmospheric Sulphur
National Research Development Corpn. *BP* 819,782

Cellulosic materials, e.g. tissue paper or cotton cloth, that have been impregnated or coated with a chromate and/or phosphate of copper adequately prevent silver, copper or other metals wrapped in them from becoming tarnished by atmospheric sulphur. C.O.C.

Flame and Heat Resistant Fabrics containing Asbestos and Cotton
Thermoid Co. *USP* 2,884,343

The fabric is impregnated with a mixture of a flame-retarding agent, plasticiser, water, and an inert texture-imparting agent, e.g. mica. The latter not only assists in imparting the flame-retardant finish but also maintains the original flexibility and smoothness of the fabric. C.O.C.

Metallised Textiles
G. W. Kunsch *BP* 816,906

Loose fibres, slivers or yarns are impregnated with natural or artificial resins to improve the adherence of vaporised metal subsequently applied to them or yarn or fabric formed from them. Alternatively the loose fibres, sliver or yarns are metallised and are then treated with resin or lacquer before being further processed. C.O.C.

Treating Glass Fibres with Aqueous Emulsions of Metals and Alloys
Owens–Corning Fiberglas Corpn. *USP* 2,886,470

Emulsions or dispersions of metals melting below 100°c. can be used for coating glass fibres. Thus Woods metal is put into water at > 71°c. and violently stirred in homogenising apparatus to yield an emulsion of molten metal and water. Glass fibres are then treated with the emulsion and the water dried off. This yields a uniform coating of metal particles on the fibres. This coating can if desired be fused or sintered to obtain cohesion between the metallic particles. C.O.C.

Coated Fabric
M. S. Kaplan *USP* 2,885,303

Cloth is coated with a suspension of hollow microspheroids (e.g. of resin or glass) suspended in a solution of a binder. The suspension is so applied that it is deposited only in the interstices between the yarns, deposition on the raised curved surfaces of the yarns on the fabric surface being prevented. This enables production of a light-weight heat-insulating fabric which is permeable to air and water vapour. C.O.C.

Coating Webs with Extruded Plastic Material
Lowe Paper Co. *BP* 819,606

The die is set to extrude a film wider than the substrate so that the beads (thickening of the edges of the film) do not form on the substrate. Thin, narrow belts of material to which the plastic will not adhere are placed on opposite ends of the rubber backing roller. This results in the beneficial live properties of the rubber backing being retained, the beads can be trimmed off or recovered for further use without sacrifice of substrate material, and contact between the plastic and the rubber surface to which it would adhere is avoided. C.O.C.

Alkoxysilylmethyl- or Alkoxysilylpropyl-amine as Sizes for Glass Fibres
Union Carbide Corpn. *BP* 816,056

Glass fibres which have been sized with an alkoxysilylmethyl- or alkoxysilylpropyl-amine adhere better to aldehyde condensate, epoxy and urethane heat-hardenable resins than do untreated fibres. C.O.C.

Laminated Sheet Material
Toscony Fabrics *BP* 820,375

Machinery for laminating sheet material, e.g. plastic film, to cloth, paper or other backing to produce a laminate having a washable, moisture-proof, wear-resisting surface useful in wallpaper, upholstery and other decorative purposes. C.O.C.

Bonded Fibre Fabrics
E. Elöd *BP* 816,983

Where natural or synthetic rubber is used as the bonding agent, replacement of the vulcanising agents, vulcanising accelerators, anti-oxidants, etc., by a pre- or intermediate condensate of a synthetic resin having reactive groups which can cross-link the reactive groups in the rubber yields a product which has all the desirable properties of one produced by vulcanising in the normal manner. Use of the precondensate has the advantages that there is no settling out of the dispersion, no discoloration in presence of copper and no unpleasant smell develops on heating or storing. C.O.C.

Artificial Furs
L. Rona *BP* 820,279

Animal hair is clipped from the skin without disturbing the position of the individual hairs relating to one another. They are then placed on a flat surface in their natural position and the ends of the hairs next to that surface are treated so that they form a continuous felted layer which replaces the skin. Thus "second-clip" wool fleeces are placed with their cut inner side on an endless belt having convergent side guides that tend to pack the fibres more closely together, each fibre being kept perpendicular, e.g. a vacuum below and/or compressed air above the belt. The ends of the fibres next to the belt are then hot scoured under pressure. The fleece is then transferred to a brass belt which is heated. This causes the wet alkaline ends of the fibres to curl. They are fed on to another endless belt where the curled ends are treated with hot soap solution and a brush having rapid vertical reciprocating movements of < 1 mm. These heating and felting operations are repeated in succession until the desired felted layer is obtained at the bottom of the fleece. It is then sprayed with dilute sulphuric acid, care being taken that the acid does not penetrate to the base of the fleece, and is dried, baked, washed by sprays of hot water directed against the felted base of the fleece and dried by light pressure between heated rollers. Drying is completed in a hot air chamber and the felted surface is sprayed with an aqueous solution containing formaldehyde and hydrogen peroxide while being again gently pressed between hot rollers. If desired the felted base is coated with rubber or a synthetic resin and finally the fleece is carded to erect the fibres which have been flattened in previous operations. C.O.C.

Vaporised Metal Coating of Flexible Substrates (I p. 546)
Reducing the Water Imbibition of Regenerated Cellulose Threads (VI p. 567)
Modifying the Structure and Properties of Addition Polymers (VI p. 567)
Dyeing and Finishing Blends of Dacron Polyester Fibres and Cellulose Fibres (VIII p. 570)
Increasing the Wash Fastness of Disperse Dyes on Cellulose Acetate (VIII p. 571)
Flock Patterns on Textiles (IX p. 572)
Vulcanising Aqueous Dispersions of Rubber (XIII p. 577)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Transparency and Reflectance of Papers; Theory and Measurement
K. G. Schmidt *Papier*, **12**, 141–149 (1959); *Chem. Abs.*, **53**, 12672 (10 July 1959)

A new type of measurement, based on measuring against an absolutely black background and so avoiding the errors caused by differences in a "white" background, was used to determine the transparency, opacity, and brightness of 12 different papers. The results were compared with Kubelka–Munk data. From the former equations were deduced by extending the mathematical approach from a single sheet to a pile of sheets, and taking account of intermediate reflectances. These new equations are simpler than those obtained by the Kubelka–Munk analysis but give very similar results. The possible interdependence of optical properties and sheet thickness is briefly discussed. C.O.C.

Accessibility of Various Cellulose Preparations from Jute

D. K. R. Choudhury

Text. Research J., **29**, 394–397 (May 1959)

Results of viscosity measurements in cuprammonium hydroxide soln. of various jute cellulose preparations and the materials hydrolysed in 2·4 N-HCl at 100°C. under N_2 enabled the levelling-off DP and hence the crystallite length to be calculated. The effect of hemicellulose present in the preparations on the accessibility and levelling-off DP was studied. S.B.D.

PATENTS

Paper Filled with Synthetic Resin

W. S. Hodgkinson & Co. *BP* 820,236

Paper suitable for producing decorative and wear-resistant laminates is obtained by adding a water-insoluble melamine monomer to a fibre pulp slurry, forming a smooth, close-surface paper from the slurry, printing the paper and then treating it with formaldehyde or hexamethylene tetramine to produce a melamine–formaldehyde precondensate in the paper. The amount of melamine used is $< 25\%$ of the weight of the resin-filled paper. In use the resin-filled paper is hot pressed into a base material. This not only bonds the paper to the base but also effects final curing of the resin. C.O.C.

Mineral-coating Compositions for Paper

Rohm & Haas *USP* 2,885,374

Mineral-coating compositions containing starch either as the whole or part of the binder are much improved in respect to their printability by addition of hexamethylenetetramine. C.O.C.

Hydroxyethyl Cellulose Sheets

Rayonier *BP* 817,809

Modification of *BP* 782,842. An aqueous solution of an alkali metal oxide is forced through a continuously-moving sheet of dry and fibrous natural cellulose. After excess of the lye has been removed ethylene oxide is passed through the moving sheet which is finally washed free of residual caustic, dried and calendered. The treatment is such that 0·1–2·0% ethylene oxide on the weight of the cellulose is taken up. The sheet retains its fibrous character but is denser and stronger and has improved colour and printability. C.O.C.

Giving Polyethylene Affinity for Printing Inks

DuP *USP* 2,878,519

Treating polyethylene with H_2O_2, HNO_2, alkaline hypochlorite, conc. HNO_3 or a mixture of conc. HNO_3 and conc. H_2SO_4, at 25–90°C. renders polyethylene adherent to printing inks without impairing the transparency. C.O.C.

Vaporised Metal Coating of Flexible Substrates (I p. 546)
Cellulose-alkali-biuret-copper (or Nickel) Complexes (VI p. 567)
Impregnated Cellulosic Materials for Preserving Metals Susceptible to Atmospheric Sulphur (X p. 575)
Coating Webs with Extruded Plastic Material (X p. 575)
Laminated Sheet Material (X p. 575)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Model for the Helix–Random Coil Transformation in Polypeptides

L. Peller *J. Phys. Chem.*, **63**, 1194–1206 (July 1959)

(1) *The Model and its Thermal behaviour.* A partition function is used to describe configurations of a long polypeptide chain in aqueous solution. A thermal transition from a helical to a random coil configuration may occur. The temperature at which the transition occurs depends on the difference in nearest neighbour interactions and entropy differences between amino acid residues in the two forms.

(2) *Influence of Solvent Composition and Charge Interactions on the Transition.* Increasing activity of a second component interacting preferentially with random coil residues causes a transition to a random coil. The helix-coil transition in a polyglutamic acid should result from increasing the pH and the transition is accompanied by considerable ionisation of COOH groups in a very narrow pH range. The possible significance of this type of ionisation for anomalous titration behaviour of some proteins is discussed. W.R.M.

PATENTS

Softening Skins and Hides

Boehme Fettchemie *BP* 818,132

Hard skins which are difficult to soften are treated with mercaptoalkane sulphonic acids (alkane of 2–4 C), or their salts, e.g. triethanolamine salt of 1,4-mercaptobutane-sulphonic acid. This softens the skins without causing undesirable swelling and can be carried out at a pH which does not favour bacterial growth. A firm leather of good handle and grain is obtained. C.O.C.

Tanning Agents

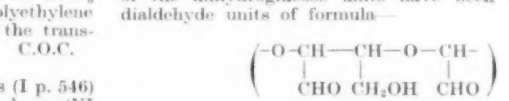
BASF *BP* 817,101

Thoroughly mixing together or heating in presence of water (*a*) a tanning salt of a polyvalent metal and a strong mineral acid, capable of forming a complex and dispersible in water with an acid reaction; (*b*) an aliphatic, aromatic, aliphatic–aromatic or heterocyclic compound containing at least one primary, secondary, tertiary or quaternary amino group or an acid amide group, the molar ratio of $a : b$ being 1 : 0·4–5·0; (*c*) an aldehyde, the weight ratio $(a + b) : c$ being 1 : 0·1–6·0; (*d*) a phenolic compound of the benzene or naphthalene series containing at least one OH bound to the nucleus, the weight ratio $(a + b) : d$ being 1 : 0·2–1·0; and (*e*) a condensable methylol compound etherified on the methylol hydroxide, the weight ratio of $(a + b) : e$ being 1 : 1·0–6·5, yields a tanning agent having amphoteric properties and containing little neutral salt. Thus urea, butylene diamine, and benzylamine are slowly added to aq. $AlCl_3$. Pyrogallol followed by HCHO are then added and the whole heated for 30 min. at 90°C. Glyoxal is then added followed by heating for 60–120 min. The solution is brought to pH 6·5–6·8 with soda ash and a solution of the dimethylol compound of butane-diol-diurethane added. The resulting stable dispersion is used with either anionic or cationic fat liquors, tanning being completed at pH 5–6 by adding formic acid after the fat-liquoring is completed. C.O.C.

Tanning with Periodate Oxypolysaccharides

U.S. Secretary of Agriculture *USP* 2,886,401

Periodate oxypolysaccharides when used as tanning agents yield smooth-grained, light-coloured leather. They are formed by treating a polysaccharide, e.g. starch, dextrin or cellulose with periodic acid so that $< 10\%$ of the anhydroglucose units have been converted to dialdehyde units of formula—



C.O.C.

Coated Leather

Rohm & Haas Co. *USP* 2,879,178

When coating leather with aqueous dispersions of polymers much better adhesion is obtained when the polymers contain amino groups. Thus leather treated with an aqueous dipersion of the copolymer of ethylacrylate and 2-aminoethyl vinyl ether (95 : 5) pigmented with Red Iron Oxide (C.I. Pigment Red 101) is given a fairly adherent coating whereas one based on poly(ethyl acrylate) alone is readily stripped off.

USP 2,880,116

Similar effects are obtained if the polymer contains t-amino groups and (1) the aqueous vehicle contains either an epoxide condensate and/or (2) a lacquer-type coating composition containing in solution an epoxide condensate is subsequently applied to the coating containing t-amino groups.

USP 2,884,336

The leather is first coated with an aqueous dispersion containing polyvalent metal ions and a water-insoluble copolymer of monoethylenically unsaturated monomeric units 3–12% of which contain COOH groups, e.g. a dispersion of a copolymer of acrylic acid and an acrylic ester and basic aluminium acetate. It is then dried at a temperature which causes the copolymer particles to coalesce to form a coating on the leather. Finally a coating of a film-forming addition polymer in an organic solvent is applied, e.g. a vinylchloride–vinylacetate copolymer in a

mixture of xylol, methyl and ethyl ketones, and ethoxyethylacetate. It is preferable that any colorant to be used shall be included in the aqueous dispersion. This yields coated leather of excellent softness and flexibility with a colour of good fastness to rubbing.

USP 2,884,340

Five different two-coat systems for producing leather of the above character are described. C.O.C.

Water-repellent Finish for Leather

Dow Corning Corpn. *USP* 2,884,393

A mixture of (1) 15–50% by wt. of a compound of formula—

$$\left[\begin{matrix} R^1C\text{–}O\text{–} \\ \| \\ R^2C \\ | \\ R^2CO \end{matrix}\right]_n ZrY_{4-n}$$

(R^1 = aliphatic hydrocarbon; R^2 = H or aliphatic hydrocarbon; R^2 = aliphatic hydrocarbon or aliphatic hydrocarbonoxy; n = 0·75–4·0; Y = O or aliphatic hydrocarbonoxy), e.g. a partly condensed octyl zirconate, (2) 5–70% of a resin made up of trimethylsiloxy and SiO_2 units so that $CH_3 : Si :: 1·0–1·5 : 1$ and (3) 5–80% of a polysiloxane of former $Z_mSiO_{\frac{4-m}{2}}$

(Z = hydrocarbon of < 7 C; m = average value of 2·0–2·9) used as a water-repellent finish yields leather which can be readily coated with standard leather finishes. C.O.C.

Coating Casein Films with Cellulose Nitrate Lacquers

FBy *BP* 816,704

Addition of formamide to cellulose nitrate lacquers considerably improves their adhesion to casein films even when the latter have been hardened. Such lacquers are particularly suitable for improving the resistance to water, fastness to wet rubbing, and the gloss of leather dressed with a pigmented casein finish. C.O.C.

Vaporised Metal Coating of Flexible Substrates (I p. 546)

XIII— RUBBER; RESINS; PLASTICS

Colouring of Plastics

A. Haehl *Chim. et Ind* (Paris), **81**, 535–543 (1959): *Chem. Abs.*, **53**, 14573 (10 July 1959)

A review of the mineral and organic pigments used to colour the following types of plastics: phenolic, amino, glyceorophthalic, polyester, polyethylene, polystyrene, poly(vinyl chloride), cellulose acetate, acetobutyrate, poly(methacrylic esters), and polyamides. C.O.C.

PATENTS

Finishing of Dipped Rubber Goods

London Rubber Co. *BP* 815,825

Treatment with a liquid alkyl siloxane as described in *BP* 769,184 before vulcanisation not only results in goods of enhanced polish and transparency but prevents surface oxidation during vulcanisation. C.O.C.

Vulcanising Aqueous Dispersions of Rubber

Revertex *BP* 816,230

Exposure to radiation of 1–60 megarads causes rubber to become crosslinked without the use of any chemicals. The crosslinks so formed are more stable to heat than those of sulphur vulcanisates. C.O.C.

Cellulosic Coatings for Vinyl Copolymers

Eastman Kodak Co. *USP* 2,887,397

Vinyl copolymers are coated with a lacquer containing cellulose acetate butyrate dissolved in a solvent mixture of ethylacetate, isobutylacetate, ethylalcohol, toluene, and dimethylformamide. This yields a coating which prevents plasticiser migrating from the vinyl polymer when in contact with other surfaces which may absorb plasticiser and which imparts high gloss. Addition of colorants to the lacquer enables single or multiple colours to be produced on the polymer surface. C.O.C.

Chlorosulphonated Polyethylenes as Smooth, Tough and Non-blocking Films and Coatings

B. F. Goodrich Co. *BP* 816,867

Compositions comprising a curable chlorosulphonated polyethylene are rendered capable of being readily dried if they are blended by hot mixing or fluxing with a polyethylene. The mixture with the addition of the necessary curing agent and pigments can be dissolved in volatile organic solvents at room temperature and used to produce non-blocking films or coatings. C.O.C.

Patterned Material

Beck, Koller & Co. (England) *BP* 816,927

A polyester resin is applied in a pattern to a base sheet and allowed to harden. This is followed by application of another coating of polyester resin which contrasts visually with the first coating. A composition of glass fibre and polyester resin is applied on top of the two coatings and after it is hardened the base sheet is stripped off. C.O.C.

Applying Flock to the Surfaces of Irregularly-shaped Articles

London Rubber Co. *BP* 815,834

The articles, e.g. rubber or plastic gloves, are coated with adhesive and passed through a chamber in which a cloud of flock is maintained. The cloud is formed by passing air into the chamber through a container in which flock is being agitated. Unused flock is withdrawn from the chamber and returned to the container. C.O.C.

Rendering Polyethylene Surfaces Adherent to Hydrolysed Vinyl Chloride–Vinyl Acetate Copolymers, Polyvinylalkyl Ethers, and Acrylic or Methacrylic Polyesters

Union Carbide Corpn. *USP* 2,879,177

Polyethylene surfaces after being treated with chlorosulphonic acid, washed with water and dried have good adherence for inks or coating compositions based on hydrolysed vinyl chloride–vinyl acetate copolymers, polyvinylalkyl ethers or polyesters of acrylic or methacrylic acid and an aliphatic monohydric alcohol. C.O.C.

Irradiated Filler-containing Polyethylene

General Electric Co. *BP* 820,168

Irradiated filled polyethylene in which the filler is silica or a metal silicate has better flexibility, tensile strength, elongation, tear impact, and flexural strength than irradiated unfilled polyethylene and better high temperature properties than either irradiated unfilled or irradiated filled polyethylene where other fillers have been used. C.O.C.

Preventing Discoloration of Chlorosulphonated Polyolefin Polymer Coatings

B. F. Goodrich Co. *USP* 2,881,098

One or more coats containing chlorosulphonated polyolefin polymer are applied and then vulcanised. One or more additional coats containing the polymer are subsequently applied and vulcanised. This prevents materials in the substrate which cause the polymer to discolour migrating into the top layers of the coating. C.O.C.

Metallised Plastic Films

Commonwealth Engineering Co. *USP* 2,884,337

The heated film is subjected to an atmosphere of a compound which at the temperature of the film decomposes to deposit metal on the film. Thus cellulose acetate film at 280° F. when treated with nickel carbonyl at atmospheric pressure becomes coated with nickel. The metallised side has a mirror finish but viewed through the other side the film is transparent. C.O.C.

Decreasing the Hydroxyl Number of Polyesters

Lonza Electric & Chemical Works *BP* 815,843

Treatment with diketone much reduces the hydroxyl number of polyesters. C.O.C.

Vaporised Metal Coating of Flexible Substrates (I p. 546)
Ultraviolet-absorptive Additives in Lacquers and Plastics (V p. 564)
Flame-resistant Acrylonitrile Polymers (VI p. 567)
Modifying the Structure and Properties of Addition Polymers (VI p. 567)
Coating Shaped Polymers with a Non-polymerisable Compound (X p. 573)
Bonding Polyethylene Tereplithalate Fibres to Rubber (X p. 574)
Coating Webs with Extruded Plastic Material (X p. 575)
Laminated Sheet Material (X p. 575)
Giving Polyethylene Affinity for Printing Inks (XI p. 576)

XIV— ANALYSIS; TESTING; APPARATUS

Photometric Titrations

J. B. Headridge *Talanta*, **1**, 293–304 (1958): *Chem. Abs.*, **53**, 5951 (10 April 1959)

A comprehensive review of recent applications of this technique to analytical chemistry. Photometric titrations are classified as: (1) titrations in self-indicator systems, (2) titrations in solutions containing more than enough of an indicator to complex either the substance being titrated or post-endpoint additions of titrant, (3) titrations in solutions containing sufficient indicator to complex only a portion of the substance being titrated or of post-endpoint additions of titrant. C.O.C.

Electronic pH Measurements under Extreme Conditions. III— Measuring pH in Non-aqueous Liquids

K. Schwabe *Chem. Ing. Tech.*, **31**, 109–117 (1959): *Chem. Abs.*, **53**, 8777 (25 May 1959)

Review, over 50 references. C.O.C.

Centrifugal Force in Paper Chromatography and Electrophoresis

H. J. McDonald, L. P. Ribeiro, and L. J. Banaszak *Anal. Chem.*, **31**, 825–829 (May 1959)

A description of the apparatus and technique of centrifugally accelerated paper chromatography. Also of an apparatus for the additional application of an electrical field at right angles to the centrifugal field. An example, using amino-acids, shows how the R_f values are slightly increased and the development time considerably reduced, when compared with the conventional descending method. P.B.S.

Artifacts in Paper Chromatography

G. Zweig *Anal. Chem.*, **31**, 821–824 (May 1959)

Artifacts (substances formed from the sample material during purification or chromatography) causing multiple spots in the chromatography of amino-acids, organic acids and bases, 2,4-dinitrophenylhydrazones, and inorganic ions are discussed. Their identity and methods of minimising their occurrence are given. P.B.S.

Separation and Identification of Peroxides by Paper Chromatography

J. Cartlidge and C. F. H. Tipper *Chem. and Ind.*, (26), 852–853 (27 June 1959)

Determination of Hypochlorites with Rhodamine B (C.I. Basic Violet 10)

R. L. Dremlyuk and N. M. Stasho *Trudy Odessk. Gosudarst. Univ. im. I. I. Mechnikova, Ser. Khim. Nauk*, **146**, (5), 55–58 (1956): *Chem. Abs.*, **53**, 8941 (25 May 1959)

A solution of Rhodamine B standardised with NaClO is acidified with 5 ml. N-H_2SO_4 and titrated with the solution of ClO^- from a microbiuret until the colour disappears. It is possible to determine 0·026 mg. or more of hypochlorites with a maximum relative error of about 1·66% for a 0·01 N-solution and 1·25% for a 0·001 N-solution. The procedure is simple, takes only 3–5 min. and is unaffected by presence of chlorates. C.O.C.

Standardisation of Testing Methods for Auxiliaries— XIV

K. Hintzmann and W. Langmann *Melliand Textilber.*, **40**, 552–554 (May 1959)

The dispersing power of surface-active agents is estimated by the darkening produced in a zinc oxide–soot mixture. 10 g. ZnO and 0·5 g. soot are treated in a specified ball mill together with 5 to 10 ml. of a 1% solution of surface-active agent and 25 ml. of water for 15 min., the mixture then being painted on to white cardboard and compared with a ten-part grey scale standard. Darkening varies linearly with specific surface of soot particles, which is itself a function of concentration for any given dispersing agent. The relationship breaks down for excessive milling times or concentrations of dispersing agent, as, when all the soot has been dispersed, comminution of the harder ZnO particles begins to occur. S.M.J.

Standardisation of Testing Methods for Auxiliaries— XV

H. Bundesmann *Melliand Textilber.*, **40**, 555–556 (May 1959)

An account of preliminary work designed to carry out scouring trials, to test efficiency of scouring, using a German Standard apparatus for assessing foaming power. The perforated disc therein is replaced by a 26 mm. high tin with perforations not only on upper and lower, but also on side surfaces. Eighteen layers of test fabric 35 mm. in diameter are used. Difficulties include uneven wash effect and interference by foam. S.M.J.

Some Alkyl-Aryl Sodium Sulphonates as Standard Substances in Detergent Chemistry

E. Q. Laws and W. Hancock *Nature*, **183**, 1473–1474 (23 May 1959)

The product of mol.wt. and optical density of $CHCl_3$ soln. of the methylene blue cpd. is approx. constant for *n*-octylbenzenesulphonate and its higher homologues. These data, and similar data for other similar cpd., are given. The relationship affords a useful check on the mol.wt. of unknown cpd. of similar composition. The R_f values for octylbenzenesulphonate and three higher homologues in various mixtures of alcohol and water are given. J.W.D.

Separate Determination of Non-ionic Surfactants in Presence of Ionic Agents

M. Kurata and S. Inoue *Yukagaku*, **6**, 92–96 (1957)

Assay of Both Non-ionic Surfactant and Quaternary Ammonium Salt in the Mixture

Ibid., 96–100: *Chem. Abs.*, **53**, 7630 (25 April 1959)

Anionic agents (Na sperm alcohol sulphate or Na alkylbenzenesulphonate) or an ionic agent (9-octadecen-1-yl-picolinium chloride (I)) were added to 0·07–0·3% solution of higher alcohol ethers of polyoxyethyleneglycol (II). To 3 meq. of such mixture 8 meq. of ion-exchange resin was used at a flow rate of 0·14–0·22 c.c./c.c. resin/min. The non-ionic agent was determined in the effluent. Accuracy was 96·8 ± 2·5%. The mixture of I and II or dodecyltrimethylammonium chloride could be separately determined by combining known methods, e.g. the ferricyanide and the Epton methods or the ferricyanide and the bromophenol blue methods. C.O.C.

Tragacanth Solutions. II— Determination of Thickening Capacity and Stability

G. Levy and T. W. Schwarz *Drug Standards*, **26**, 153–156 (1958): *Chem. Abs.*, **53**, 8535 (10 May 1959)

Procedures are outlined for determining thickening capacity, stability and the effect of other substances on the stability. C.O.C.

Determination of the Ratio of Methyl to Phenyl Groups in Silicone Polymers

J. H. Lady, G. M. Bower, R. E. Adams, and F. P. Byrne *Anal. Chem.*, **31**, 1100–1102 (June 1959)

Some of the polymer is smeared on a sodium chloride plate, and the solvent allowed to evaporate. The infrared absorption of the film is then measured at two wavelengths. The absorption at 6·97 μ. is due to phenyl–silicon linkages, and that at 7·9 μ. to the methyl–silicon linkages. The ratio of these absorbances gives the phenyl/methyl ratio from the calibration curve, which is prepared using standard polymers made from methyl- and phenyl-chlorosilanes, since carbon–hydrogen analysis is subject to many errors. P.B.S.

Differentiation of Dyes

S. Nishiyama and H. Ebara

I— Paper Chromatography of Red and Orange Dyes

Kagaku to Sōsa, **11**, (2), 45–48 (1958)

11 Different types of red dyes and 4 of orange were dissolved in CH_3OH–H_2O (7 : 3) as a developing solvent. Most of the dyes had each their own characteristic R_f value and fluorescence. The method has the advantages of requiring shorter developing time, good reproducibility, quick drying and stability of dyes during development.

II— Absorption Spectra of Red and Orange Dyes

Ibid., 49–58: *Chem. Abs.*, **53**, 7599 (25 April 1959)

Xanthene dyes have characteristic absorption curves in the ultraviolet region which enable them to be distinguished from other types of dye. Those showing the change in λ_{max} in the visible range gave corresponding change in λ_{max} in the ultraviolet by treatment with acid or alkali. Reduction of dyes causes disappearance of their characteristic absorption curves. Except for

xanthene dyes differentiation of dyes from their absorption curves in the ultraviolet and visible ranges is difficult. C.O.C.

Reversed-phase Chromatography. II— R_f Values of Solvent Dyes on Silicone-treated Paper
M. R. Verna and R. Dass
J. Sci. Ind. Research (India), **17B**, 304–306 (1958): *Chem. Abs.*, **53**, 8913 (25 May 1959)

R_f values are given for solvent dyes separated on silicone-treated paper by using mixtures of water, alcohol, and hydrocarbons or halogeno hydrocarbons as developing agents. C.O.C.

Identification of Organic Pigments by Paper Chromatography, Some Applications
Z. Moloster *Ann. chim.* (Paris), [13], **3**, 771–814 (1958): *Chem. Abs.*, **53**, 8949 (25 May 1959)

Previous work by others is surveyed and the techniques used for this work described. Several dyes from each of the following 13 groups were investigated as 0·1–0·25% solutions in water or alcohol: nitroso, nitro, azo, pyrazole, diphenylamine, triphenylmethane, xanthene, acridine, quinoline, thiazole, oxazine, thiazine, and azine. For each dye the Schultz and C.I. 1st Ed. No. are given, the solvent used as developer, time of development, R_f value and the colour of the resultant spots examined under various conditions. The R_f value for a given dye was independent of its source. Diagrams show the separation of members of the same group of dyes. The technique can be used to identify dyes in lipsticks and inks. 53 references. C.O.C.

Coulometric Titration of Dyes with Electrolytically Generated Dithionite
M. Munemori *Talanta*, **1**, 110–116 (1958): *Chem. Abs.*, **53**, 5951 (10 April 1959)

Dithionite, electrolytically generated at a Hg pool cathode in an electrolytic cell containing HSO_3^-, was used to titrate Methylene Blue (C.I. Basic Blue 9) and Indigo Carmine (C.I. Acid Blue 74). By titrating at room temperature equilibrium was quickly established. The end point was determined photometrically. Optimum pH range for titration was 3–5 and the concentration of HSO_3^- was 0·01 M. Very small samples (mg.) were determined with an average error of 0·5%. C.O.C.

Analysis of Hair Dyes
C. J. Turi

III— Observations and Remarks on the Chromatography of Aromatic Amines
Rend. ist. super. sanità, **21**, 748–758 (1958)

A description of a microchromatographic unit which can be used for two-dimensional chromatography and is rapid enough to prevent oxidation of the sensitive amino compounds. Tails and comets are reduced by adding a drop of Na hydrosulphite (which does not react with the amino compounds) or by a short boil of the test solution with activated C and filtering before the test. Diamines give two spots (one of the salts, the other of the hydrolysed base) in strong acid solution, but only the base spot when the chromatography is done in weak acid (acetic) solution. Ascending R_f values for the common materials met in hair dyes are given for butanol–acetic acid–water 4 : 1 : 5 (Solvent A) and butanol–ethanol–water 4 : 1 : 5 (Solvent B). Additional spot test tables are also given.

IV *Ibid.*, 759–785: *Chem. Abs.*, **53**, 7514 (25 April 1959)

The systematic analysis of hair dyes has been elaborated to include more recent developments such as use of aminosulphonic acid and the separation of dyes from other components of hair preparations. The emollients are most easily removed by acidifying with HCl and filtering (with or without adding NaCl); this was superior to various methods of column chromatography. The resulting aq. HCl extract contained all the dyes ready for paper chromatography. Two-dimensional chromatography of 11 commercial samples is described. The acid extract was neutralised with ammonia to free the bases. C.O.C.

Identification of Amines in Hair Dyes by Paper Chromatography
J. Deshusses and P. Desbaumes
Mitt. Gebiete Lebensmitt. u. Hyg., **49**, 335–343 (1958): *Chem. Abs.*, **53**, 9580 (25 May 1959)

The amines were extracted from the acidified hair dyes with ether and then treated with 4 different reagents (vanillin in HCl, p-$(CH_3)_2NC_6H_4CHO$ in N-HCl, 0·1 N. $AgNO_3$–NH_3, 1% acetaldehyde in acetic acid–ethylalcohol) after ascending chromatography with Whatman Paper No. 1. Solvents used were a butanol-conc. HCl–water (70 : 20 : 40 by vol.) and 2% NH_4OH saturated with butanol. In 14 commercial hair dyes the following amines were found: m- and p-tolylenediamines (C.I. Oxidation Base 20 and C.I. 76042 respectively), m-phenylenediamine (C.I. Developer 11), dimethyl-p-phenylenediamine (C.I. Oxidation Base 8), m- and p-aminophenols (C.I. Oxidation Base 7 and 6 respectively), methyl-p-aminophenol, diaminophenol, and aminodiphenylamine. Their R_f values are given. C.O.C.

Spectrophotometric Determination of Dyes in Hydrophobic Colour Images
Y. N. Gorokhovskiĭ and V. P. Kupchinskaya
Trudy Leningrad. Inst. Kinoinzhener., (3), 213–219, (1955): *Referat. Zhur. Khim.*, Abst. No. 44749 (1956): *Chem. Abs.*, **53**, 6848 (25 April 1959)

A spectrophotometric method for determining the surface concentration of dyes in 3- and 4-component hydrophobic colour images is described. Possible sources of errors during the determination were investigated and limits of the method's practical application established. C.O.C.

Paper Chromatography of Synthetic Dyes authorised for use in Food in the Argentine— I
R. C. T. Puche *Publs. inst. microquím., Univ. nacl. litoral* (Rosario, Arg.), **21**, (23), 134–146 (1957): *Chem. Abs.*, **53**, 8462 (10 May 1959)

Two-dimensional chromatography (solvents: butanol saturated with 10% aq. HCl; mixture of 0·5 ml. xylidine, 5 ml. 33·53% aq. HCl and 10 ml. water) was performed with the following dyes in 0·3% aqueous solution Erythrosine (C.I. Food Red 14), Rose Bengal, Red Burdeos, Ponceau 2R (C.I. Food Red 5), Orange I (C.I. Acid Orange 20), Sunset Yellow (C.I. Food Yellow 3), Indigotin (C.I. Food Blue 1), Naphthol Yellow S (C.I. Food Yellow 1), Tartrazine (C.I. Food Yellow 4), Acid Violet 6B, and Guinea Green B (C.I. Acid Green 3). Results and R_f values are given in tables. C.O.C.

Detection of Metanil Yellow (C.I. Acid Yellow 36) in Sweetmeats Rich in Casein
S. N. Mitra and S. C. Roy
Sci. and Culture, **23**, 202–203 (1957): *Chem. Abs.*, **53**, 8465 (10 May 1959)

The method for detecting Metanil Yellow previously described (*Chem. Abs.*, **51**, 14151 (1957); J.S.D.C., **74**, 75 (1958)) is unsuitable for sweets rich in casein. A modified procedure using warm 90% alcohol is described. C.O.C.

Green Pigmentation of Olive Oils
F. De Francesco *Boll. lab. chim. provinciali* (Bologna), **9**, (1), 90–95 (1958): *Chem. Abs.*, **53**, 8467 (10 May 1959)

The spectrophotometer is unreliable for testing for traces of chlorophyll in H_2SO_4-treated oils but the spectrofluorimeter gives accurate results. C.O.C.

Detecting and Distinguishing between Butter Yellow (C.I. Solvent Yellow 2) and Somalia Yellow 2G (C.I. Solvent Yellow 1) in Edible Oils and Fats
L. Polzella *Boll. lab. chim. provinciali* (Bologna), **9**, (1), 96–99 (1958): *Chem. Abs.*, **53**, 8468 (10 May 1959)

Dissolve 20 ml. oil or 20 g. fat in 40 ml. ether in a separating funnel. Add 3 mol. of 5% $SnCl_2$ in conc. HCl, shake, add 25 ml. water and shake. Separate the aqueous layer and filter into a second funnel. Neutralise to phenolphthalein with 20% NaOH, agitate and extract with two 10-ml. portions of $CHCl_3$. Filter the extracts, evaporate to 3 ml., add 0·5 N-HCl, shake, add 3–4 drops H_2S solution, shake and add 1 drop 10% $FeCl_3$. Violet (thione or Louth's violet) indicates C.I. Solvent Yellow 1 and blue (Methylene Blue) indicates presence of the carcinogenic Butter Yellow. The test is accurate to 0·5 mg./100 g. oil or fat. C.O.C.

Chromatographic Identification of Pigments in Wine and its Adulterants— I
R. C. T. Puche
Publs. inst. invest. microquím., Univ. nacl. litoral (Rosario, Arg.), **21**, (23), 129–133 (1957): *Chem. Abs.*, **53**, 8531 (10 May 1959)

Pigments and Vitamins. III— Paper-chromatographic Separation and Determination of Carotenoid Pigments
Y. Yamamoto and Y. Tomita
Mem. Fac. Agr. Kagoshima Univ., **3**, 16–24 (1957): *Chem. Abs.*, **53**, 8313 (10 May 1959)

Partition chromatography using impregnated paper is a rapid and simple method for separating and determining the carotenoid pigments in plants. Both CuO and Mg papers gave 95% recovery with such pigments as carotene (C.I. 75130), lycopene (C.I. 75125) and lutein. The results obtained agree well with those from the A.O.A.C. column chromatographic method. The minimum detectable quantities are 0·05 γ and 0·04 γ for carotene and lycopene respectively. The determinable minimum for carotene is 10·2 γ%, and for lycopene is probably the same. C.O.C.

Improvements in Paper Chromatography Technique for Sap-soluble Plant Pigments
K. Lesins and I. Lesins
Proc. Genet. Soc. Can., **3**, 44–46 (1958): *Chem. Abs.*, **53**, 8322 (10 May 1959)

A method for separating sap-soluble anthocyanins and anthoxanthins which takes only 4–6 hr. C.O.C.

Spectrophotometric Determination of Quercetin
L. E. Dowd *Anal. Chem.*, **31**, 1184–1187 (July 1959)

The sample is dissolved in isopropyl alcohol and diluted with water. A portion containing 4–5 mg. quercetin is then diluted with standardised aluminium chloride reagent (0·01 M. adjusted to pH 4·0) and left to stand 10 min. for the colour to develop. The absorption is determined at 430 mμ. using a 1 cm. cell.

The method was developed for quercetin from Douglas fir bark (separated by water extraction) and gives a standard deviation of $\pm$ 0·4%. Phlobaphenes and tannins present as impurities do not interfere. P.B.S.

X-Ray Powder Diffraction Data of Azoic Coupling Components
J. Schnopper, J. O. Broussard, C. L. LaForgia
Anal. Chem., **31**, 1542–1549 (Sept. 1959)

A sample, recrystallised from xylene, is powdered and packed into a sample holder. Using an X-ray diffractometer with automatic strip-chart recorder a diffraction pattern is obtained of intensity against diffraction angle. The interplanar spacings are calculated from the line positions and the intensities relative to the most intense line. Data is given for 15 compounds as the interplanar spacings corresponding to the four most intense lines and the largest spacing in the pattern. This data allows positive identification of 15 azoic coupling components in either impure commercial products or mixtures of two compounds. P.B.S.

Analysis of Food Dyes by Fluorescence
M. Popovici
Rev. ind. aliment., Produse vegetale, (7–8), 19–24 (1958): *Chem. Abs.*, **53**, 11682 (25 June 1959)

A study of the fluorescence shown by 165 synthetic dyes when irradiated by ultraviolet radiation of 3650 A. using a Philora HPW type quartz lamp, 75 w. The radiation was screened through a black NiO glass. The dyes on various substrates were placed 10 cm. from the lamp and at 45° incidence. Fluorescence was observed at right angles to the incident radiation. Some dyes show strong fluorescence, some commercial dyes with similar trade names show similarly coloured fluorescence and some chemically related dyes show dissimilar fluorescences because of impurities in them. Dyes can be identified by fluorescence if the same method of analysis is consistently used. 37 references. C.O.C.

Chromatographic Research on Artificial Dyes in Foods
V. Sadini
I— Chromatography on a Column or on Plates
Chimica (Milan), **35**, 15–12 (1959)
II— Paper Chromatography
Ibid., 81–98: *Chem. Abs.*, **53**, 11681 (25 June 1959)
Review, 273 references. C.O.C.

Colouring Cheeses with Annatto (C.I. Natural Orange 4) and Carotene Dyes (C.I. Natural Yellow 26) and the Detection of Such Dyes
G. Schwarz, H. Mumm, and F. Woerner
Molkerei- u. Käserei-Ztg., **9**, 1430–1433 (1958): *Chem. Abs.*, **53**, 11694 (25 June 1959)

Three varieties of cheese were coloured with Annatto, emulsifiable Carotene, and powdered Carotene and the keeping properties of these dyes compared. They can be detected by extracting the minced cheese (25–50 g.) for 20 hr. with acetone, evaporating off the solvent from the extract, extracting the residue with C_6H_6 (15 ml.). This solution is dried with Na_2SO_4 and poured into a silica column. Elution brought out the Carotene which was determined by Lagoni and Wortmann's method (*Chem. Abs.*, **51**, 8863 (1957)). Any Annatto present was left on the adsorbent and was detected by eluting with $CHCl_3$ and adding several drops of Carr–Price reagent to the silica when the reddish yellow Annatto ring turned bluish green. C.O.C.

Paper Chromatography and Paper Electrophoresis of Organic Materials. II— Separation and Determination of Orange I and Orange II (C.I. 14600 and 15510) by Ionisation Paper Chromatography
H. Ojima *Aichi Gakugi Daigaku Kenkyū Hōkoku, Nat. Sci.*, (7), 21–24 (1958): *Chem. Abs.*, **53**, 11112 (25 June 1959)

Orange I can be separated from Orange II by paper chromatography using as developer Kolthoff's buffer solution (0·03 M-KCl + 0·03 M-KOH, 0·03–0·1 N-NaOH or 0·05 M-Na_2CO_3). Only Orange I is ionised at the resulting pH 8·5–9·0 and it has a greater R_f than Orange II. C.O.C.

Chromatography of Dye Intermediates. IX— Paper-chromatographic Analysis of Naphthalenesulphonic Acids
J. Latinák *Collection Czechoslov. Chem. Communs.*, **24**, 922–929 (1959): *Chem. Abs.*, **53**, 13118 (25 July 1959)

Naphthalene mono-, di-, and trisulphonic acids and H_2SO_4 occurring in sulphonation mixture were separated and determined quantitatively by ascending chromatography on Whatman No. 4 paper with 2 : 1 propanol–aq. NH_4OH. Detection was done by heating the chromatogram to 200°C. or by fluorescence (caused by impurities) in ultraviolet radiation. The following R_f values were found for naphthalene-1- and -2-sulphonic acids; -1,5-, -1,6-, -1,7-, -2,6-, -2,7-, and -1,3-disulphonic acid; -1,3,6-, -1,3,5-, and -1,3,7-trisulphonic acids, SO_3^{2-}, Cl^-, NO^-, Na^+, K^+, Ca^{2+}, Ba^{2+}, Mg^{2+}: 0·75, 0·75, 0·53, 0·53, 0·53, 0·53, 0·53, 0·66, 0·36, 0·36, 0·36, 0·16, 0·49, 0·60, 0·30, 0·22, 0·23, 0·11, 0·00. Separation of naphthalenemono-, -di-, and -trisulphonic acid isomers is more difficult and descending chromatography using 2 : 1 propanol–aq. NH_4OH or 30:10 : 2 : 8butanol–ethanol–aq. NH_4OH–water is necessary. Naphthalene-1,3,6-trisulphonic acid was separated from mixture with 1,3,5- and 1,3,7-isomers by continuous development chromatography using 2 : 1 propanol–aq. NH_4OH. C.O.C.

Determination of Glass Colorants (Determination of Chromium)
N. A. Tananaev and L. I. Ganago
Trudy Ural. Politekh. Inst., Sbornik, **57**, 73–75 (1956): *Referat. Zhur., Khim.* Abstr. No. 30995 (1957): *Chem. Abs.*, **53**, 12615 (10 July 1959)

Determination of Glass Colorants (Determination of Cobalt)
N. A. Tananaev and L. I. Ganago
Trudy Ural. Politekh. Inst., Sbornik, **57**, 5–8 (1956): *Referat. Zhur., Khim.* Abstr. No. 31006 (1957): *Chem. Abs.*, **53**, 12614 (10 July 1959)

Orcein Dyes (IV p. 558)
Transparency and Reflectance of Papers; Theory and Measurement (XI p. 575)

XV— MISCELLANEOUS

Colorants for Lipsticks and Nail Varnish (IV p. 561)
Applying Flock to the Surfaces of Irregularly-shaped Articles (XIII p. 577)

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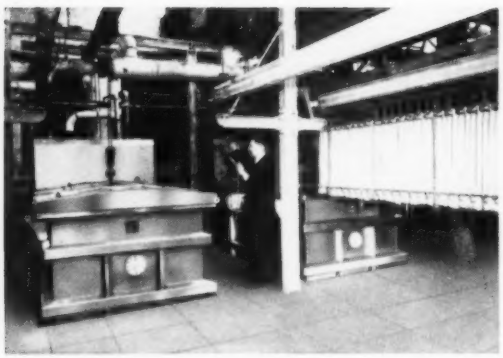
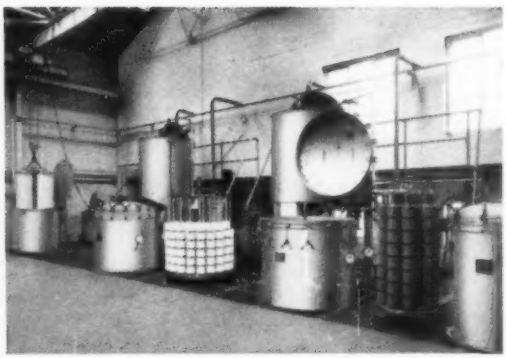


Hank Dyeing machines incorporating the Brown/Longclose double stick system (patent)



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FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page* x

Tuesday, 16th February 1960

LEEDS JUNIOR BRANCH. *Diffusion Processes in Wool Keratin.* Dr. J. A. Medley (Wool Industries Research Association). The Lecture Theatre, Department of Colour Chemistry and Dyeing, the University, **Leeds 2.** 3.30 p.m.

SCOTTISH SECTION. *The Keratin of Wool.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Joint meeting with the Textile Institute.) The Royal College of Science and Technology, **Glasgow.** 7.30 p.m.

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Thursday, 25th February 1960

WEST RIDING SECTION. *New Work on the Dyeing of Nylon with Disperse Dyes.* H. Seaman, Esq., (I.C.I. Ltd.). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Tuesday, 1st March 1960

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Friday, 4th March 1960

LONDON SECTION. *Recent Developments in Leather Dyeing.* J. H. Sharphouse, Esq. (Northampton College of Technology). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 8th March 1960

NORTHERN IRELAND SECTION. *The Dyeing and Finishing of Fabrics Containing Tricel.* R. J. Mann, Esq., B.Sc., F.S.D.C. (Courtaulds Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

HUDDERSFIELD SECTION. *Paper Chromatography of Dyestuffs.* J. C. Brown, Esq., B.Sc., A.R.I.C. (CIBA Clayton Ltd.). (Joint meeting with the Huddersfield Section of the Royal Institute of Chemistry.) Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

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Thursday, 14th April 1960

MANCHESTER JUNIOR BRANCH. Annual General Meeting. Title of paper to be announced later. T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C., Room J/E 11, Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

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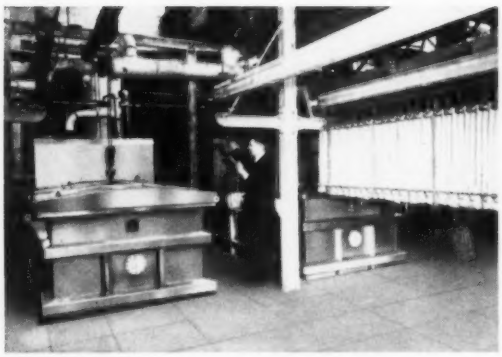

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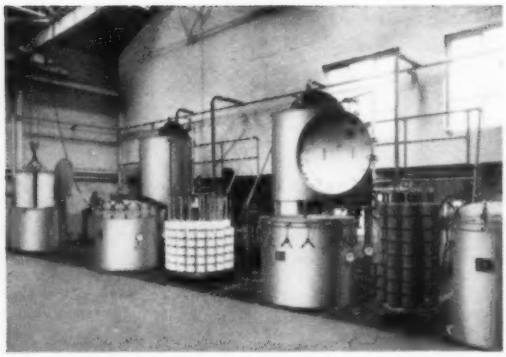

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Advertisements relating to APPOINTMENTS VACANT, APPOINTMENTS WANTED, and MISCELLANEOUS ITEMS are invited for insertion on this page. Advertisements of Appointments Wanted are gratis to members, but must not exceed twenty-four words.

All inquiries relating to Advertisements should be addressed to THE GENERAL SECRETARY, THE SOCIETY of DYERS and COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed BOX—, THE SOCIETY of DYERS and COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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The Society offers a salary of not less than £1200 per annum commensurate with qualifications, age and experience. Membership of a joint superannuation scheme is available.

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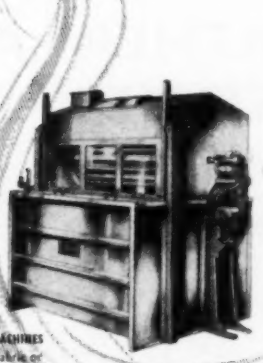

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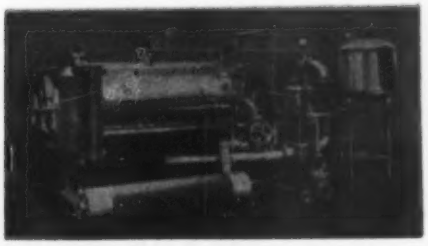

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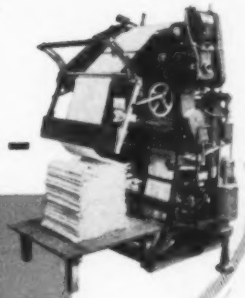

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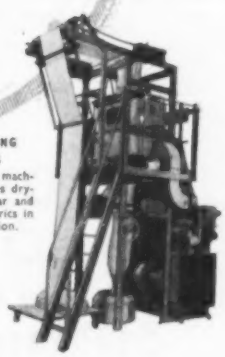

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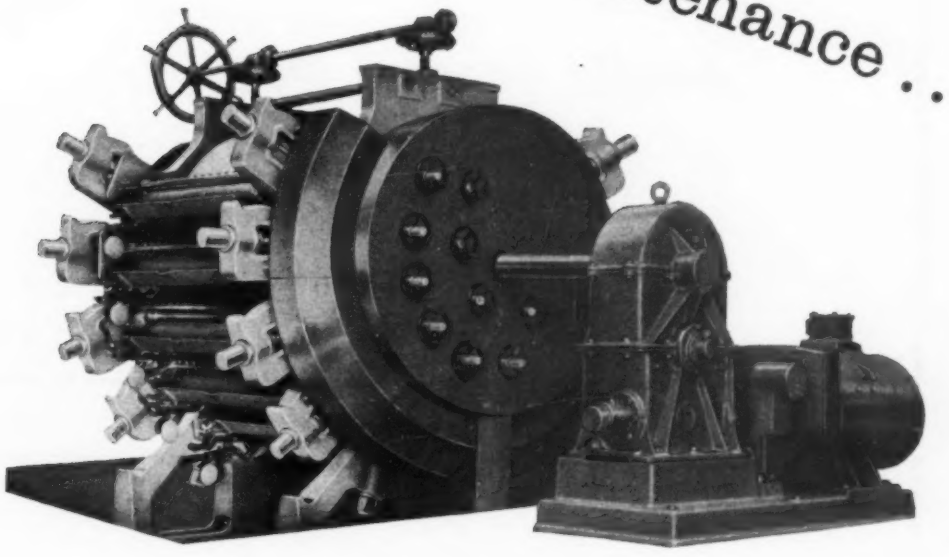

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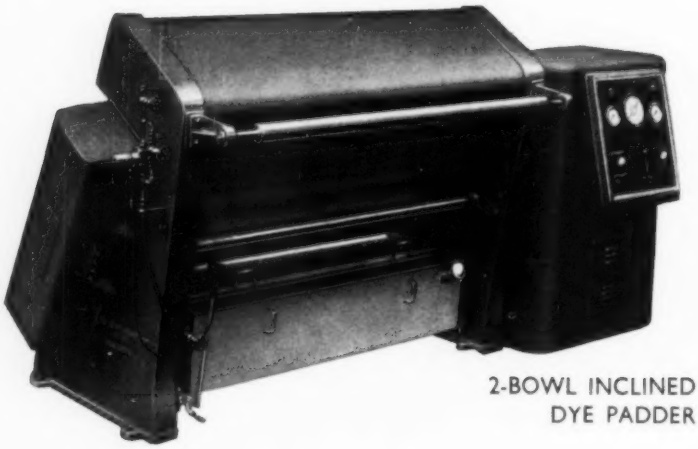

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